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Fraunhofer-Institut für Chemische  
Technologie

(1)

Investigation of the Phase  
Stabilizing Effect of Potassium  
Fluoride on Ammonium Nitrate

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## 1. INTRODUCTION

Ammonium nitrate is an interesting oxidiser in the field of propellants and explosives. The substance is cheap and readily available. The drawbacks of the substance are caused by its polymorphic properties. It crystallizes at ambient pressure in 5 modifications. Some phase transitions take place at temperatures, which occur under normal storage conditions. These transitions are connected with volume changes that influence severely the properties of products containing ammonium nitrate.

Many attempts have been made to overcome these difficulties. One of the approaches consist of incorporating different ions into the lattice to change the phase properties. In this context potassium fluoride has been used.

The doped ammonium nitrate material must be checked to make sure that no undesired phase transitions and volume changes occur in the interesting temperature interval. This is normally done by the methods of thermal analysis like DSC and DTA. These methods register thermal effects without delivering direct information on the lattice.

In the case of ammonium nitrate, however, these elaborated methods are not sufficient. A variety of phase transitions may occur, so that the phase behaviour cannot be understood, if the participating phases cannot be identified. This goal can be reached with diffraction measurements like x-ray and neutron diffraction.

A measuring system was built up in the past, which allows automatic measurements of series of diffraction patterns while cycling a sample stepwise through the interesting temperature ranges. The series contain informations about phase transition temperatures and the participating phases. The system was used to investigate the phase properties of ammonium nitrate samples, which were melted with 2 weight % of potassium fluoride. (2)

In the first interim report the preparation of the samples, DSC-measurements and preliminary series of x-ray-diffraction measurements were reported.

In the second interim report 5 series of diffraction measurements with dry samples of ammonium nitrate including documentation were covered:

<u>series</u>	<u>temperature program</u>
KF 230288	20/ 80/-70/ 80
KF 250288	20/ 80/-70/ 80
KF 230388	20/-70/ 80/-70
KF 170388	20/150/-70/ 20
KF 290388	20/-70/150/ 20

In this final report all 10 measured series are discussed. A series with humide material, which is part of the extended project, is already here included, because it should be discussed together with the dry samples.

The documentation of the additional series has been organized by means of an appendix. It contains for each series the original diffraction patterns, where each third patterns has been plotted, together with difference curves, the explanation of which is included in the appendix. Besides, informations are given about the lattice distances  $d$  and the peak intensities related with the concentration of the phases.

Additional series:

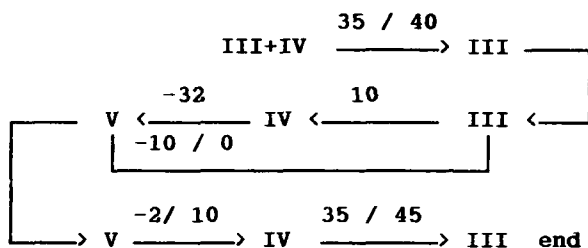
<u>series</u>	<u>temperature program</u>
KF 050988	20/ 80/-70/ 80
KF 280988	20/ 80/-70/ 80 (humide)
KF 060988	20/-70/ 80/-70
KF 140988	30/140/-70/140
KF 220988	20/-70/140/-70

## 2. RESULTS OF THE MEASUREMENTS

### 2.1 TEMPERATURE PROGRAM 20/ 80/-70/ 80

#### 2.1.1 SERIES KF 230288

At room temperature the sample contains phase III with a small amount of Phase IV. The phase changes during the temperature program can be summarized as follows:



At the onset of the heating cycle the minor amount of phase IV disappears so that at 40 °C the whole sample consists of phase III. No further changes are observed on heating.

On cooling phase III changes into phase IV beginning at 10 °C. The transition III/IV not yet completed, surprisingly, the residue of III changes directly into phase V. After -10 °C peaks of phase III were no longer detected. Starting at -32 °C phase IV changes into phase V as expected. However, a small amount of the sample remains in phase IV even at the lowest temperatures indicated by a weak (111)<sub>IV</sub>-peak.

During the following heating cycle phase V changes completely into phase IV in the range of -2 to +10 °C, followed by a complete transition IV → III between 35 and 45 °C.

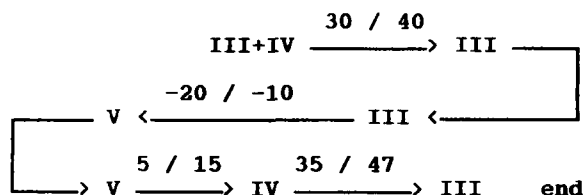
The described transitions are visible in the difference curve and can also be followed in the diffraction patterns

by means of the peaks with the assigned indices. The documentation of the series is included in the appendix of the second interim report.

#### 2.1.2 SERIES KF 250288

20/ 80/-70/ 80

At the beginning the sample consists again mainly of phase III with a small amount of phase IV. The phase changes can be summarized as follows:



Immediately after beginning the heating cycle the minor amount of phase IV disappears as in the preceding series. On cooling, phase III changes completely into phase V starting at -10 °C. This direct and complete transition III/V had not been expected. In this detail the series distinguishes itself from the preceding one.

On heating, the complete transition V/IV is followed by the complete transition IV/III.

The described transitions can be followed in the diffraction patterns and with the difference curve. Additionally, the intensity curves of the peaks (220)<sub>III</sub>, (020)<sub>IV</sub> and (022)<sub>V</sub> show nicely the appearing and disappearing phases. The intensity curves are rather rough. This is caused by the used monochromator, which provides high resolution on the expense of the intensities with the consequence of reduced measuring statistics.

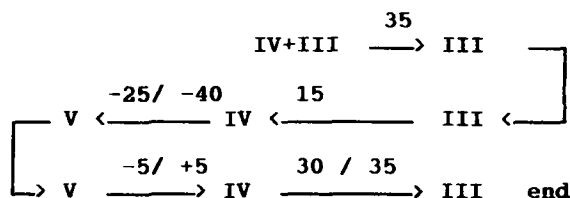
The phase changes can also be followed by the lattice plane

distances calculated from the appearing and disappearing peaks. Intensive peaks appear earlier and disappear later during the observed phase transitions.

From the lattice plane distances of the different phases the lattice parameters of the elementary cell were calculated. A reasonable fit of the lattice parameters is considered a proof for the presence of the different phases. The plots and the previously mentioned curves are included in the appendix of the second interim report.

2.1.3 Series KF 050988  
20/ 80/-70/ 80

At the beginning the sample consists mainly of phase III with a minor amount of phase IV. The phase changes can be summarized as follows:



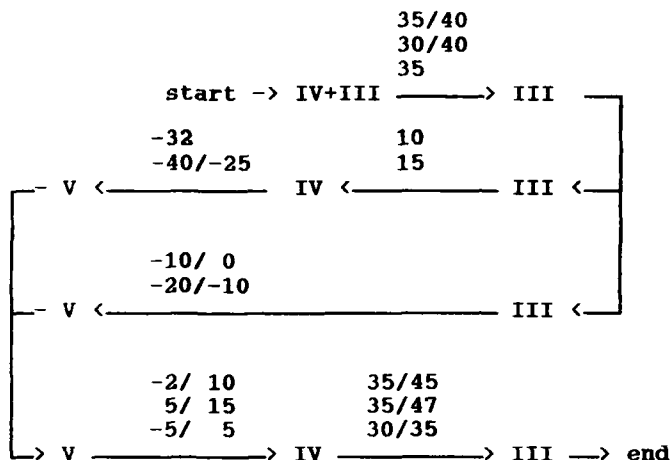
On heating, phase IV disappears until 35 °C. Phase III remains the only phase on heating and on cooling until 15 °C, when it changes completely into phase IV. On further cooling, at -25 °C, phase V appears increasing its intensity until -40 °C. After the cooling cycle the sample changes completely into phase IV from -5 to +5 °C and then into phase III from 30-35 °C.

The changes can be seen in the original diffraction patterns, in the difference curve and by the intensity curves in the appendix of this report.



#### 2.1.4 Summary of the Temperature Program 20/ 80/-70/ 80

The phase changes of the three series with the same temperature program can be summarized as follows:



At the beginning all samples consisted of phase III with a minor amount of Phase IV, which changes into phase III between 35 and 40 °C. Phase III remains the only phase on further heating.

On cooling the samples have the choice between different transitions paths. During the expected path phase III changes between 10 and 15 °C into phase IV, which undergoes a further transition into phase V between -25 and -40 °C.

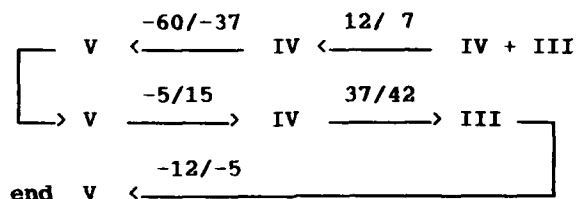
A second path includes the direct transition from phase III into phase V between 0 and -20 °C, and in a third example the direct change III/V and the sequence III/IV/V occur side by side.

During the second heating from -70 to 80 °C the results are rather homogeneous. The samples change between -5 and 15 °C from phase V into phase IV and then into phase III between 30 and 45 °C.

## 2.2 TEMPERATURE PROGRAM 20/-70/ 80/-70

### 2.2.1 Series KF 230388

In contrast to the preceding series the sample consists at room temperature mainly of phase IV with smaller amounts of phase III. The phase changes can be summarized as follows:



At the begin of the cooling cycle the small amount of phase III disappears followed by the transition IV/V. Again a small amount of phase IV is always present during the whole cooling cycle.

On heating the already known phase changes V/IV and IV/III are observed. Both transitions are completed in a relatively narrow temperature interval.

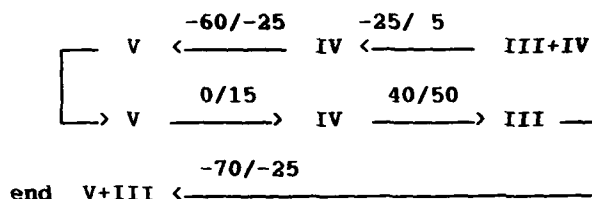
On cooling phase III changes directly into phase V in a narrow temperature interval without leaving any phase III peaks in the diffraction patterns.

The described changes can be followed easily in the diffraction patterns, whereas the difference curve doesn't show all changes clearly.

The appearing and disappearing phases can also be followed with the intensity curves, lattice distances and lattice parameters included in the appendix of the second interim report.

2.2.2 Series KF 060988  
20/-70/ 80/-70

At the beginning of the series the sample consists mainly of Phase III with a minor amount of Phase IV. The observed phase changes can be summarized as follows:



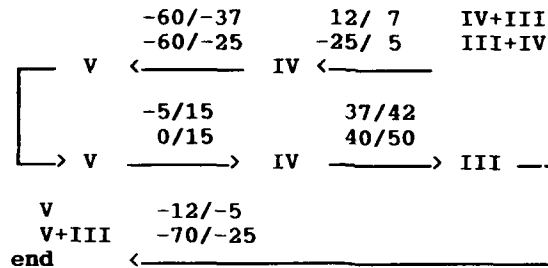
On cooling the sample changes into phase IV. At the same time, when the last peaks of phase III disappear, the peaks of phase V appear. Eventually there is a direct transition III/V. The transition IV/V is again rather sluggish.

On heating the phase changes V/IV and IV/III are more spontaneous. The temperature ranges, in which they occur, are broadened by the fact that the very first and the very last observed intensities have been included.

During the second cooling cycle phase III remains unchanged until -25 °C, when the first peaks of phase V appear. However, phase III remains the dominant phase in the last part of this cycle.

### 2.2.3 Summary of the Temperature Program 20/-70/ 80/-70

The transitions of the two series can be summarized as follows:



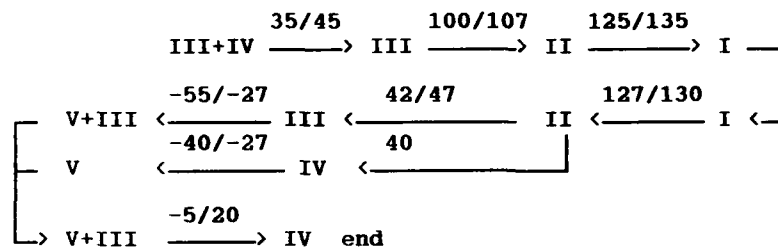
One sample consisted at the beginning mainly of phase IV and the second mainly of phase III. The transitions into phase IV started at the same temperatures. In the second series phase III remains existent down to deeper temperatures. This, however, may be due to the fact that more phase III was present in the sample at the beginning.

The differences in the other transition temperatures are not severe with the exception of the last transition III/V in the second series, where phase III remains the dominant phase even at -70 °C.

## 2.3 TEMPERATURE PROGRAM 20/150/-70/ 20

### 2.3.1 SERIES KF 170388

At room temperature the sample consisted mainly of phase III with a minor amount of phase IV. The following phase changes were observed:



On heating the peaks of phase IV are detectable until 50 °C, before it changes completely into phase III. The transition III/II begins at 100 °C before the transition II/I at 125-135 °C.

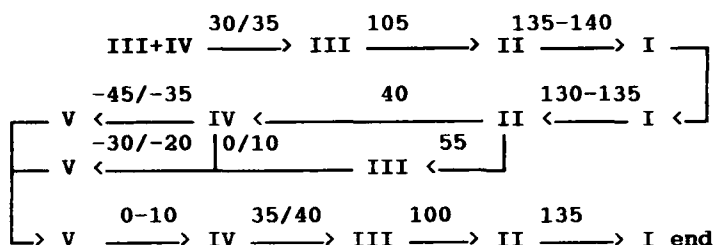
On cooling, after the change I/II a simultaneous transition II/III and II/IV occurs, which was unexpected after the former measurements. At -27 °C phase IV changes into phase V, which is considered as a normal phase behaviour. Unexpectedly, phase III is present during the whole cooling cycle. It stays on, until it decreases during the heating cycle up to 0 °C, when the change V/IV begins.

The described phase changes can be followed in the diffraction patterns, the difference curve and in the intensity and lattice parameter curves.

### 2.3.2 Series KF 140988

30/140/-70/140

At room temperature the sample consisted of phase III with a small amount of phase IV. The following phase changes were observed:



On heating the small amount of phase IV changes into phase III followed by the expected transitions III/II/I at the indicated temperatures.

On cooling phase II changes into phase III starting at 55 °C. At a temperature of 40 °C the remaining part of phase II changes into phase IV.

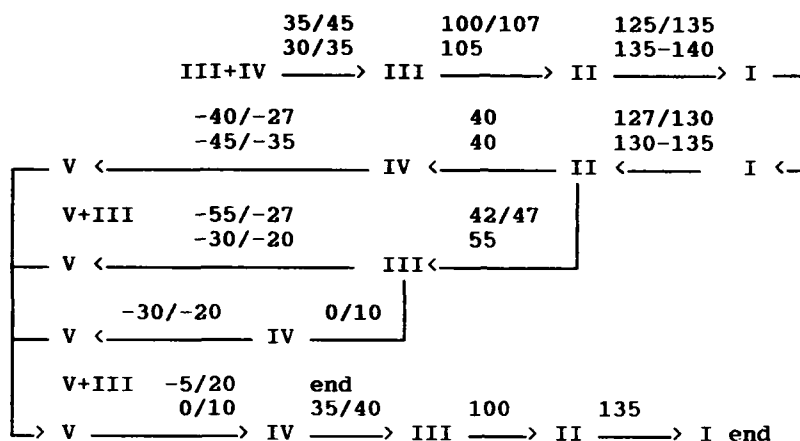
The coexisting phases III and IV follow different transition paths. Phase III changes partly into phase IV at 10 to 0 °C and directly into phase V at -20 °C, leaving no traces of phase III peaks in the patterns below -35 °C. The phase change III/IV causes an intensity increase of the Phase IV peaks.

At deeper temperatures phase IV shows the known sluggish transition into phase V.

Reheating leads to the familiar transition path V/IV/III/II/I at the known transition temperatures.

2.3.3 Summary of the Temperature Programs 20/150/-70/ 20  
and  
20/140/-70/140

The summary of the 2 series looks as follows:



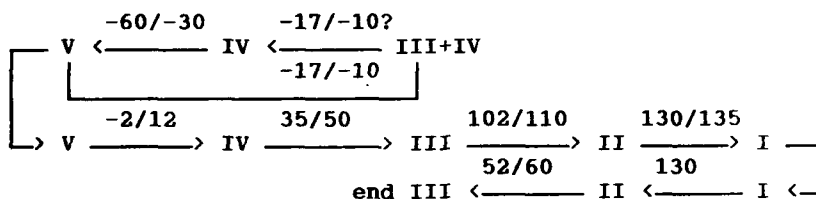
During the heating cycles the samples are rather uniform. On cooling, however, there is quite a variety of changes. The only change that doesn't occur is the change II/V, which is observed in dry pure ammonium nitrate.

The series show strong differences in this part of the temperature program, as phase III remains present in the former series even at -70 °C.

## 2.4 TEMPERATURE PROGRAM 20/-70/150/ 20

### 2.4.1 SERIES KF 290388

At room temperature the sample consisted mainly of phase IV with a minor amount of phase III. The following phase changes were observed:



On cooling the smaller amount of phase III changes simultaneously into the phases IV and V. This cannot be seen clearly in the diffraction patterns. The intensity curves, however, show a decreasing intensity of phase III and, simultaneously, an increase of phase IV and V, before the phase IV intensity decreases due to the change IV/V. Again, the (111) peak of phase IV is detectable in the diffraction patterns during the whole cooling cycle.

On heating the change V/IV occurs between  $-2$  and  $12$  °C followed by the change IV/III starting at  $35$  °C and the changes III/II and II/I at  $102$  and  $130$  °C, resp.

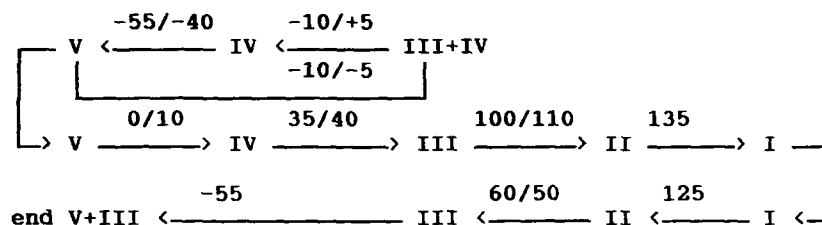
On cooling phase II reappears at  $130$  and phase III reappears at  $55$  °C. The diffraction patterns show strong texture effects after the change I/II influencing strongly the peak intensities. This effect is very often observed on cooling, when the transition temperature II/I was surpassed.



2.4.2 SERIES KF 220988

20/-70/140/-70

At the beginning the sample consists of phase III with minor amounts of phase IV. The following phase changes were observed:



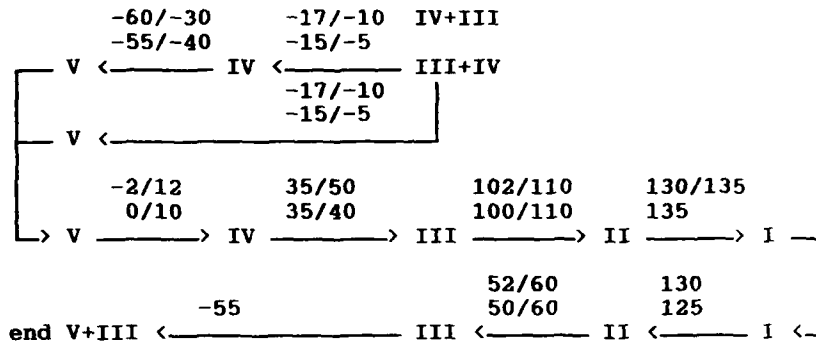
On cooling the major part of phase III changes into phase V at about -10 °C. A smaller part changes into phase IV and later between -40 and -55 °C into phase V. Below -20 °C peaks of phase III were no longer visible in the patterns.

On heating the familiar transition path V/IV/III/II/I occurs at the already known transition temperatures.

During the following cooling cycle phase IV is not observed. This, however, can also be due to texture effects that are always present, when the sample was heated above the transition II/I. Instead phase III changes into phase V at unusually deep temperatures. As a consequence the transition is not complete and the peaks of phase III are still visible at -70 °C.

2.4.3 Summary of the Temperature Programs 20/-70/150/ 20  
and 20/-70/140/-70

The transitions can be summarized as follows:



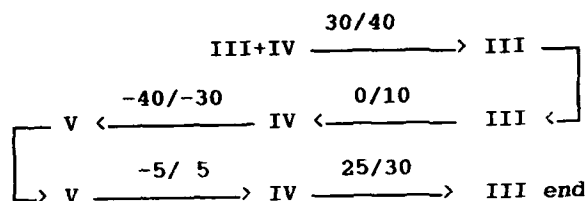
The transition paths and transition temperatures don't show essential differences, though once phase IV and then phase III prevailed in the samples at the beginning. Remarkable is the existence of phase III at -70 °C.

2.5 TEMPERATURE PROGRAM 20/80/-70/80

SERIES KF 280988

Humide Ammonium Nitrate

At the beginning the sample consists of phase III with a small amount of phase IV. The following changes were observed:



On heating the small amount of phase IV changes into phase III. on cooling phase III changes into phase IV and then into phase V as expected.

The second heating cycle shows the expected phase changes into phase IV and then into phase III.

There are no surprises with humide ammonium nitrate. The series doesn't show any of the more complicated paths especially on cooling, which are frequently observed in the dry samples.

### 3. DISCUSSION

#### Samples at room temperature

The samples had been stored for more than one week at room temperature before the measurements. They consist of a mixture of the phases IV and III. In different samples both phases can prevail.

The existence of phase III is due to the incorporation of potassium ions. The potassium concentration is not yet high enough to make phase III the only stable phase at room temperature.

#### Phase changes on heating and cooling

To get a more comprehensive view of the results, the phase changes have been summarized in table 1. The table contains all series with the different phase changes in separate columns. In this way the corresponding values in different series can be compared easily.

The phase changes II→I and I→II occur at about 130 °C with little hysteresis on cooling. The values for the phase change III→II are also rather homogeneous at about 100 °C. On cooling, there is a considerable hysteresis, so that phase III appears not earlier than 60-50 °C. Compared with pure humid ammonium nitrate the transition temperatures are increased due to the incorporated potassium ions.

The phase change IV→III occurs reliably between 35 and 40 °C without differences between the first and second heating cycle. The hysteresis is again considerable compared with the phase change III→IV starting slightly above 0 °C.

There is one deviation amongst the values in the series KF 290388, where the transition temperature is too low. In this series, however, a large amount of phase III changes directly into phase V and the small amount of the phase

change III→IV cannot be detected reliably.

The already mentioned phase change III→V occurs below 0 °C with some more deviations of the transition temperatures. In some cases phase III is even still existent at -70 °C. Obviously at rather deep temperatures the equilibrium is frozen.

The same effect can also be observed with the phase change IV→V between -30 and -60 °C, when some peaks of phase IV can still be detected at -70 °C.

So far the observed transition temperatures are comparable in the different series even with different temperature programs. It must be taken into account that the methods doesn't deliver precise transition temperatures with the applied temperature intervals of 2.5 and 5 °C between the measurements.

The results, however, deviate considerably in the observed transition paths. The expected path includes the changes V→IV→III→II→I on heating and the reverse order on cooling. On heating, all samples show the expected phase changes V→IV and IV→III at about 0 and 35-40 °C, respectively. This is also true for the transitions III→II and II/I.

The differences occur on cooling. Phase II has the choice to change into phase III and phase IV, and phase III again can change into the phases IV and V. The phase change III→V was a surprise, as this transition hasn't been reported yet in literature. This is due to the fact that most investigations of the past were made with the normal methods of thermal analysis, which do not give informations about the participating phases of a transition.

The differences can be explained with nucleation phenomena. When phase II is cooled, the transition into the expected phase III must overcome a considerable nucleation barrier, as the structure of phase III is different from phase II.

The resulting supercooling may result into the change into phase IV, which shows less supercooling due to the related structure.

In the same way nucleation may be a problem for the transition III-→IV. The resulting supercooling brings the phase III into the stability range of phase V, so that this newly observed transition becomes possible.

The transition IV-→V occurs at temperatures, where the equilibrium is already freezing. The more intensive peaks of phase IV can therefore still be detected at -70 °C.

In the series KF 170388 the corresponding effect is observed with phase III, which still shows intensive peaks at -70 °C decreasing with increasing temperature, when the region of frozen equilibrium is left on heating.

In the humide sample the picture seems to be less complicated. The presence of water eventually makes it easier to form nuclei of structurally not related phases, so that the expected transition path is chosen. The reproducibility should eventually be supported by further series.

#### 4. SUMMARY

The incorporation of 2% KF into the ammonium nitrate lattice extends the stability range of phase III. At room temperature, the samples consisted therefore of a mixture of the phases IV and III, where phase III often prevailed.

The observed transition temperatures are comparable in the different samples even with different temperature programs.

On heating, the expected transition path V→IV→III→II→I is observed. On cooling a variety of transition paths becomes visible composed of the expected transitions I→II→III→IV→V and, besides, the transitions II→IV and III→V.

The incorporated amount of 2% KF can not yet exclude a number of phase transitions under storage conditions.

**Table 1: Summarized Phase Changes**

Sample Temp. Program	Phases at 200C	P H A S E C H A N G E S									
		V<-IV	V<-III	V->IV	IV<-II	IV<-III	IV->III	III<-II	III->II	II<->I	
KF 230200 20/80/-70/80	III+IV			start	IV		35/40	III			
		V	-32		IV	10		III			
		V		-10/0				III			
		V		-2/10	IV		35/45	III	end		
KF 250200 20/80/-70/80	III+IV			start	IV		30/40	III			
		V		-20/-10				III			
				5/15	IV		35/47	III	end		
KF 050900 20/80/-70/80	IV+III			start	IV		35	III			
		V	-40/-25		IV	15		III			
		V		-5/ 5	IV		30/35	III	end		
KF 200900 20/80/-70/80 humide	III+IV			start	IV		30/40	III			
		V	-40/-30		IV	0/10		III			
		V		-5/ 5	IV		25/30	III	end		
KF 230300 20/-70/80/-70	IV+III	V	-60/-37		IV	12/ 7		III	start		
		V		-5/15	IV		37/42	III			
		end		-12/- 5				III			
KF 060900 20/-70/80/-70	III+IV	V	-60/-25		IV	-25/+5		III	start		
		V		0/15	IV		40/50	III			
		end	V+III	-70/-25				III			
KF 170300 20/150/-70/20	III+IV			start	IV		35/45	III		100/107	II 125/135 I
		V+III	-55/-27					III	42/47		II 127/130 I
		V	-40/-27		IV	40					II
		V+III		-5/20	IV	end					
KF 140900 30/140/-70/140	III+IV			start	IV		30/35	III		105	II 135/140 I
					IV	0/10		III	55		II 130/135 I
		V	-30/-20					III			
		V	-45/-35		IV	40					II
KF 290300 20/-70/150/20	III+IV	V	-60/-30		IV	-17/-10 ?		III	start		
		V		-17/-10				III			
		V		-2/12	IV		35/50	III		102/110	II 130/135 I
							end	III	52/60		II 130 I
KF 220900 20/-70/140/-70	III+IV	V	-55/-40		IV	-10/+5		III	start		
		V		-10/-5				III			
		V		0/10	IV		35/40	III		100/110	II 135 I
		end	V+III	-55				III	50/60		II 125 I



## 5. APPENDIX

- A Peak List
- B Experimental Details
- C Concept of Difference Diagrams
- D Series KF 050988
- E Series KF 060988
- F Series KF 140988
- G Series KF 220988
- H Series KF 280988

# Peaklist

Diffraction Angles of the Ammonium Nitrate  
Phases I,II,III,IV,V  
whit Chromium Radiation:  $\lambda = 2.2896 \text{ \AA}$

Peak Nr.	2*Theta (ca.)	I	(hkl) II	Phase III	IV	V
1	26.8				(001)IV	
	27.05		(001)II			(002)V
2	29.2			(101)III		
3	30.5	(100)I				
4	33.0		(110)II			(020)V
5	33.67					(200)V
6	33.8				(110)IV	
7	34.24			(111)III		
8	39.53			(120)III		
9	41.43			(210)III		
10	41.78			(021)III		
11	43.5		(111)II			(022)V
12	43.8	(110)I			(111)IV	
13	45.7			(121)III		
14	46.9				(200)IV	
15	46.9					(212)V
16	47.72		(200)II			(220)V
17	47.72			(211)III		
18	49.8					(221)V
19	50.-51.				(020)IV	
20	51.85			(220)III		
21	53.2		(210)II	(112)III		
22	53.8					(130)V
23	55.0				(201)IV	
24	55.7		(002)II			(004)V
25	56.54			(130)III		
26	57.4				(021)IV	
27	58.5					(014)V
28	59.02			(022)III		
29	60.41			(310)III		
30	60.9		(211)II	(202)III		
31	60.9				(221)IV	
32	61.2					(132)V
	61.2					(312)V
33	61.8				(012)IV	

Peaks I 2  
Peaks II 7  
Peaks III 13  
Peaks IV 9  
Peaks V 12

**APPENDIX B**  
**EXPERIMENTAL DETAILS**

X-ray radiation: chromium tube, monochromatic, primary  
monochromator

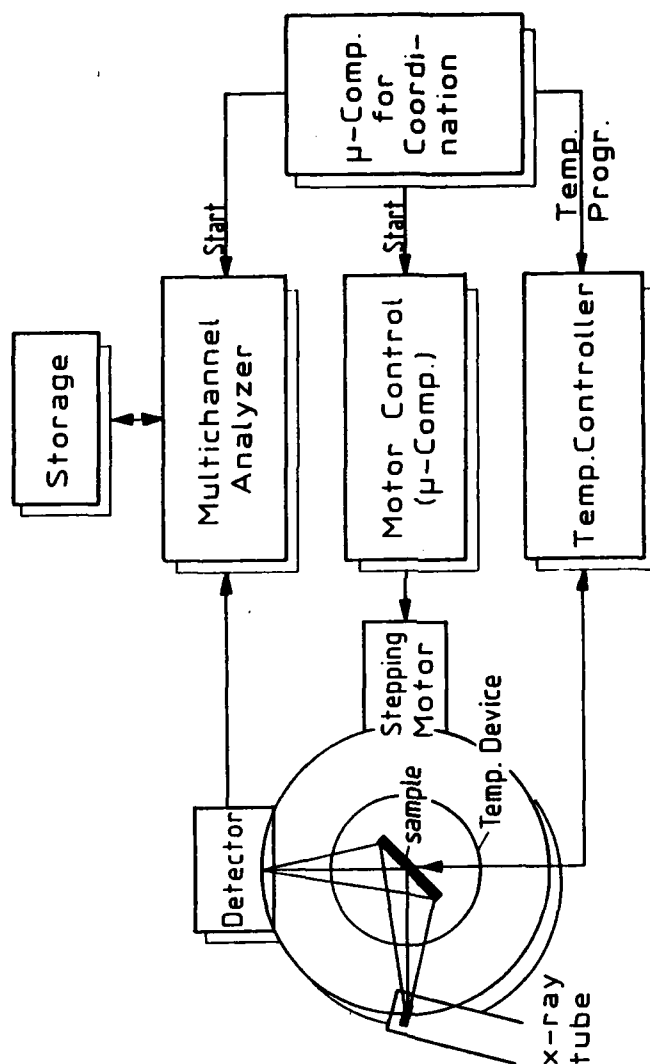
Method : angle dispersive

Detector : Position sensitive proportional counter  
(PSPD)

Angular speed : 30 and 10 °/min

Series	Temp. Program	Temp. interval °C	Angular Speed °/min	No. of patterns
KF 230288	20/ 80/-70/ 80	2,5	30	145
KF 250288	"	2,5	30	145
KF 050988	"	5	10	73
KF 280988	" (humide)	5	10	72
KF 230388	20/-70/ 80/-70	2,5	30	157
KF 060988	"	5	10	79
KF 170388	20/150/-70/ 20	2,5	30	175
KF 140988	30/140/-70/140	5	10	106
KF 290388	20/-70/150/ 20	2,5	30	175
KF 220988	20/-70/140/-70	5	10	103

## Measuring System



## APPENDIX C

### Concept of Difference Diagrams

The measuring system delivers a huge amount of data, which must be efficiently evaluated. A concept of difference diagrams was developed, which detects differences in the diagrams of a series without requiring much computing. The concept was published in J.Appl.Cryst.(1983) 16, 259-263.

The principle can be seen on the following page. If differences are formed between all corresponding channels of two patterns, a difference diagram is formed. A peak, which is present in both patterns with a slight difference of the position creates a double peak, half of which represents the positive and the other half the negative values.

If the difference is formed between two peaks with the same position, but with different intensity, the resulting difference peak has its values either in the negative or positive part.

When the absolute values of the difference formation are summed up, the sum correlates with the difference between the two patterns. If the sums between all the neighbored patterns are plotted against temperature, a curve results, which corresponds to a DSC curve (1).

When the difference formation occurs constantly between the first and all the other patterns, the corresponding curve has some similarity with a thermogravimetric curve (2).

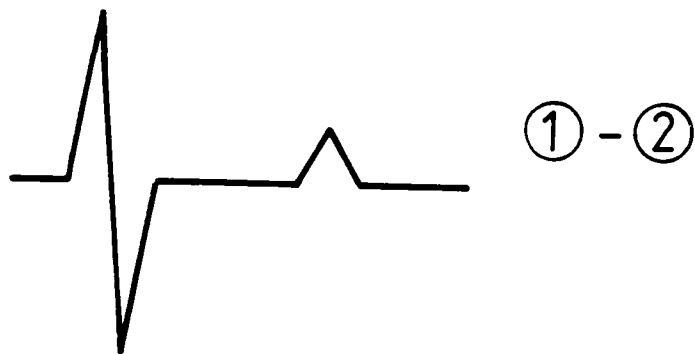
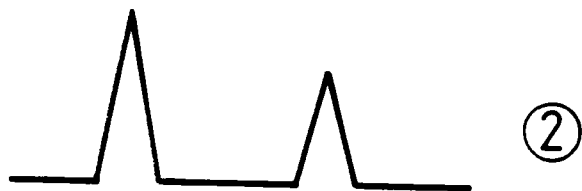
$$DY(T_i) = \sum_{j=1}^N |X_j(T_i) - X_j(T_{i-1})| ; i = 2, \dots, M \quad (1)$$

$$Y(T_i) = \sum_{j=1}^N |X_j(T_i) - X_j(T_1)| ; i = 2, \dots, M \quad (2)$$

$X_j$  is the content of the  $j$ -th channel of the  $i$ -th pattern and  $N$  is the number of channels of the pattern.  $T_i$  represents the independent variable with which the  $i$ -th pattern was recorded and  $M$  is the number of patterns in a series.

The different curves are both calculated and plotted, as some effects as phase transitions show more distinctly in the DY-curve, while thermal expansion can be seen only in the Y-curve.

# Concept of difference diagrams



*Series*  
*KF 050988*

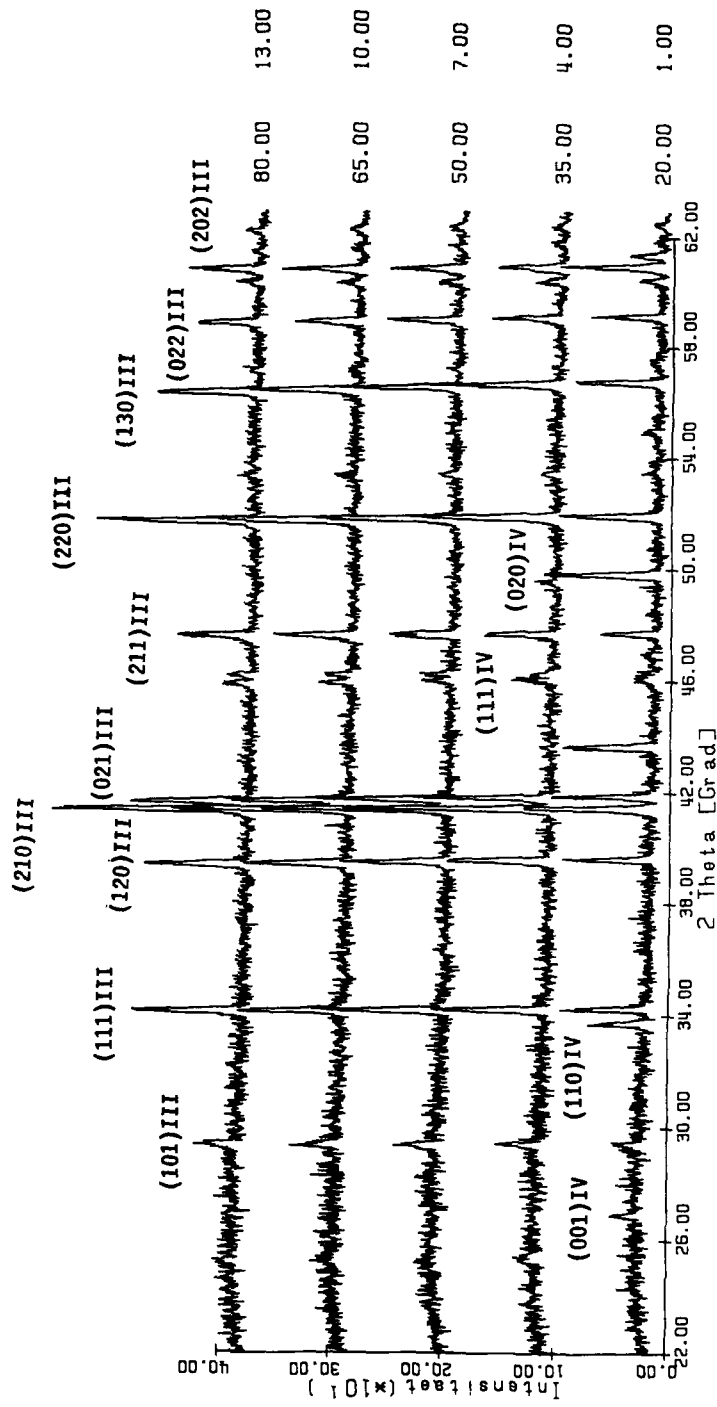
*Temperature*  
*Program*  
*20/80/—70/80*

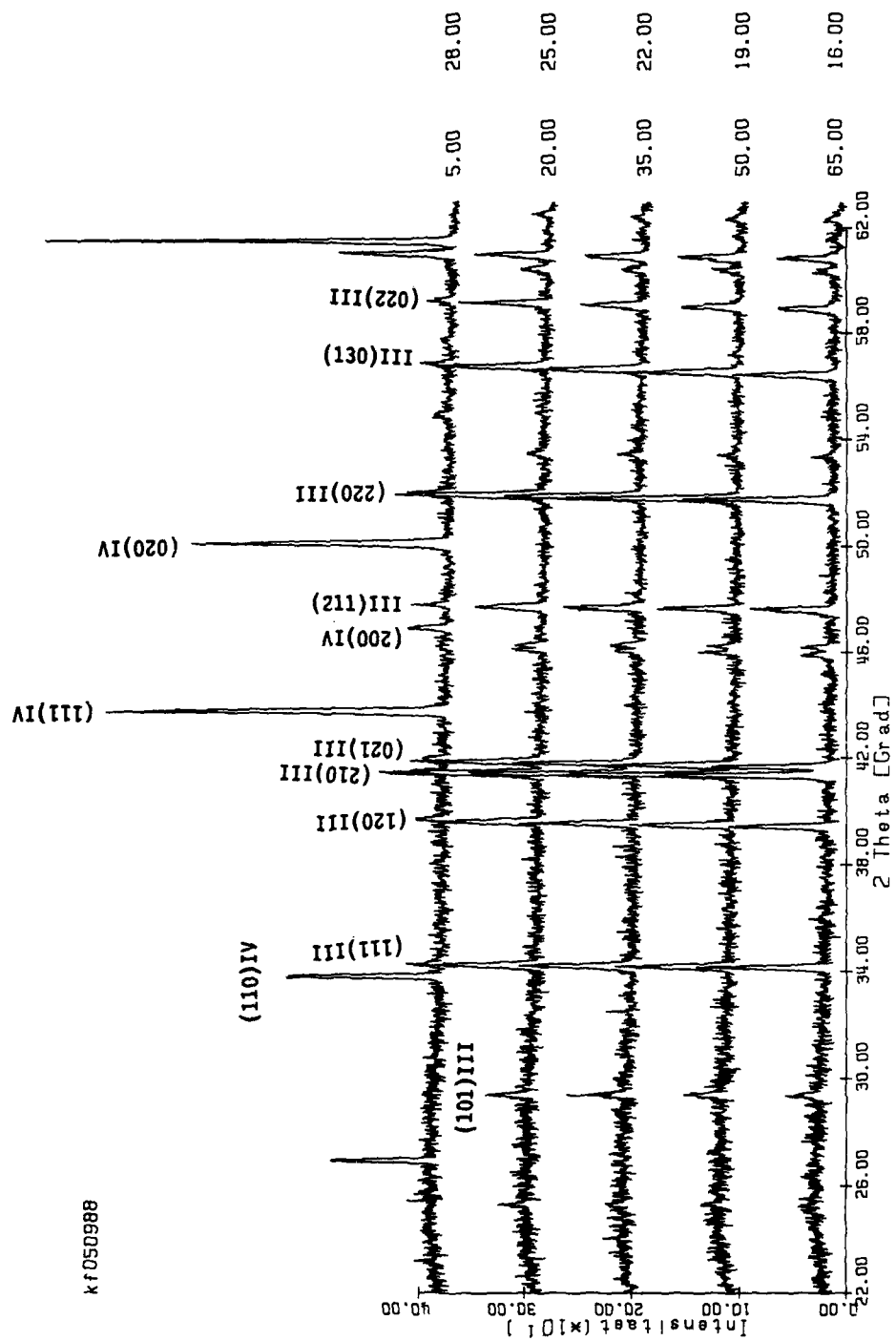


# Diffraction Patterns

kf050988

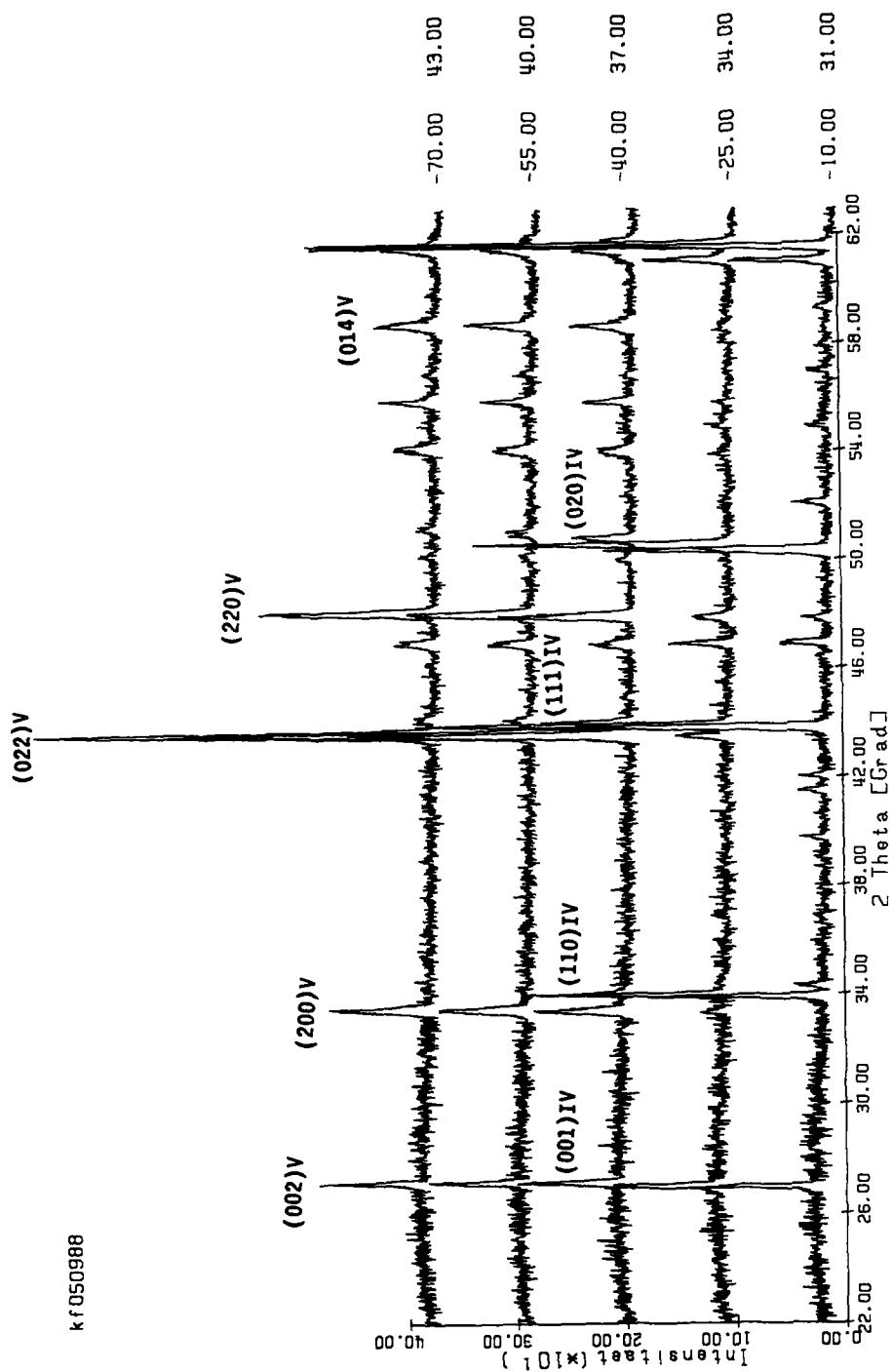
20/80/-70/80°C





k1050988

kf050988



Intensity ( $\times 10^4$ )

2 Theta [Grad]

(111)IV

(002)IV

(001)IV

(002)V

(110)IV

(200)V

(020)IV

(220)V

(004)V

(014)V

5.00

-10.00

-25.00

-40.00

-55.00

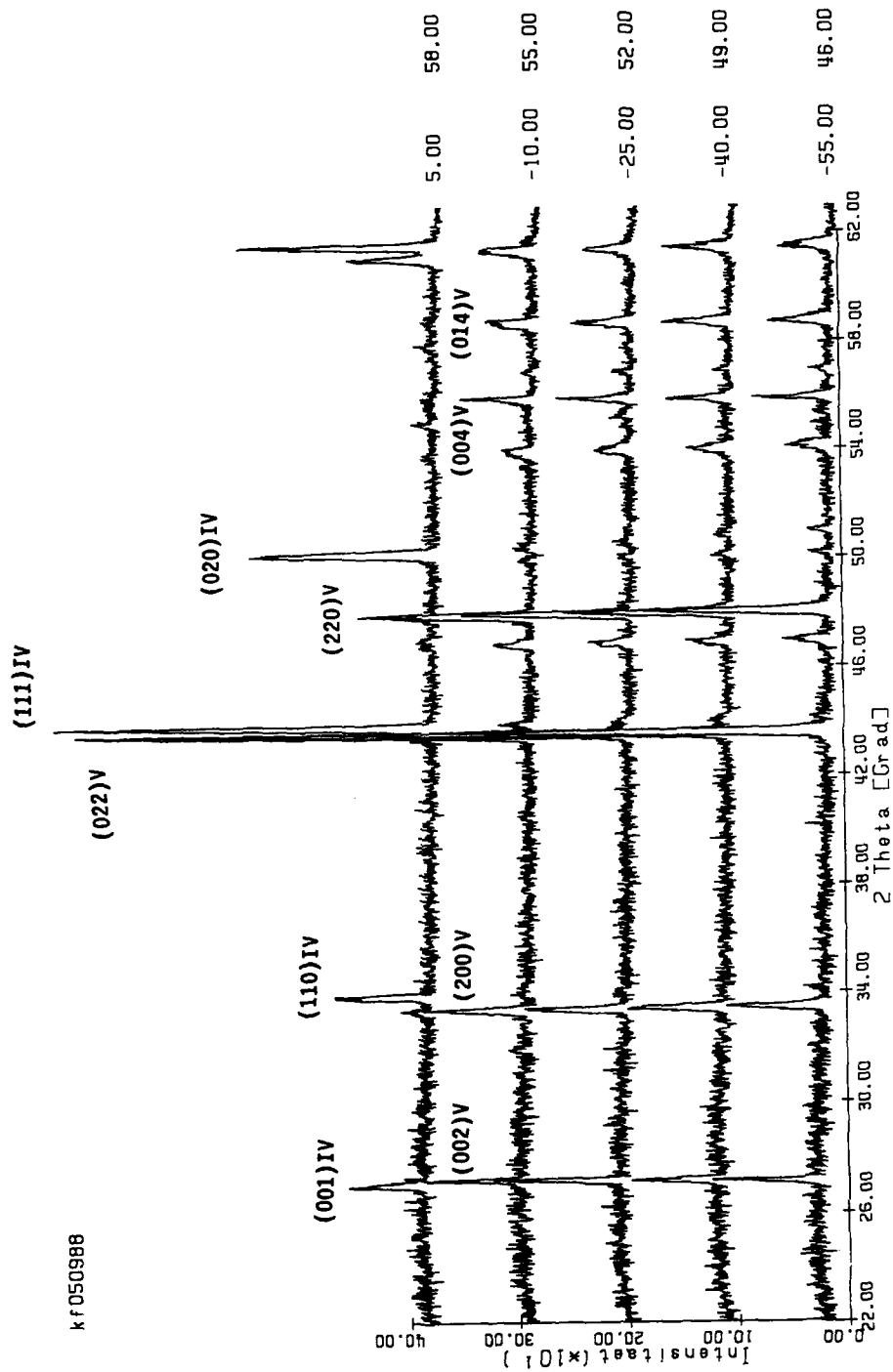
46.00

49.00

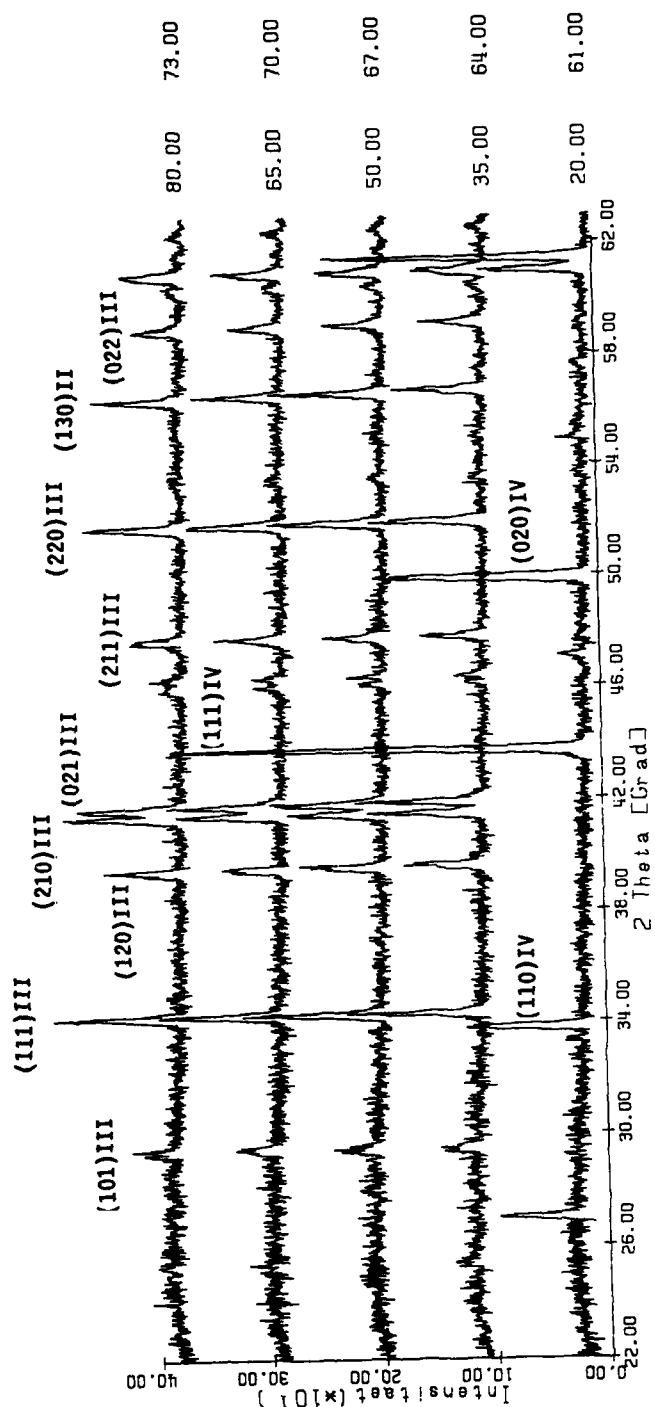
52.00

55.00

58.00



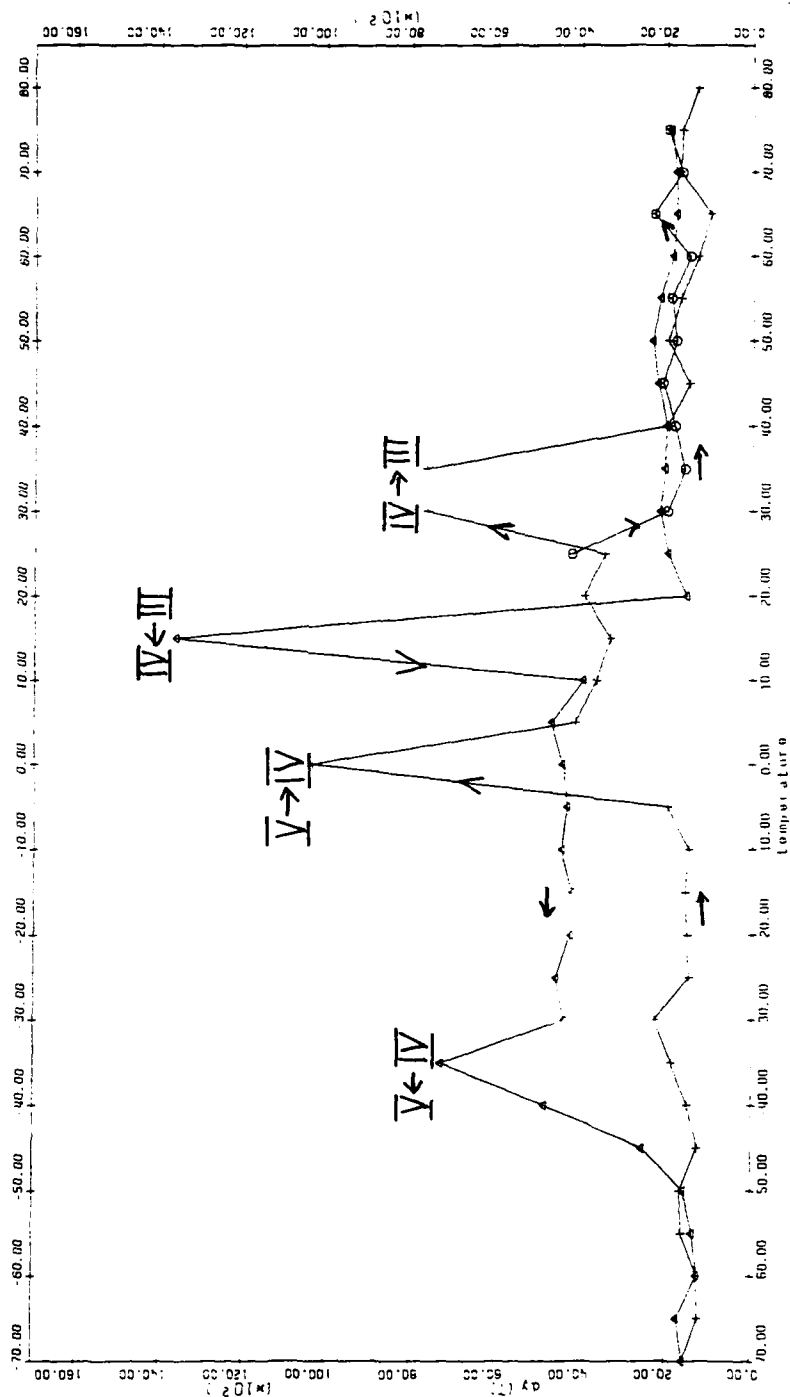
kf050988



# Difference Curve DY(T)

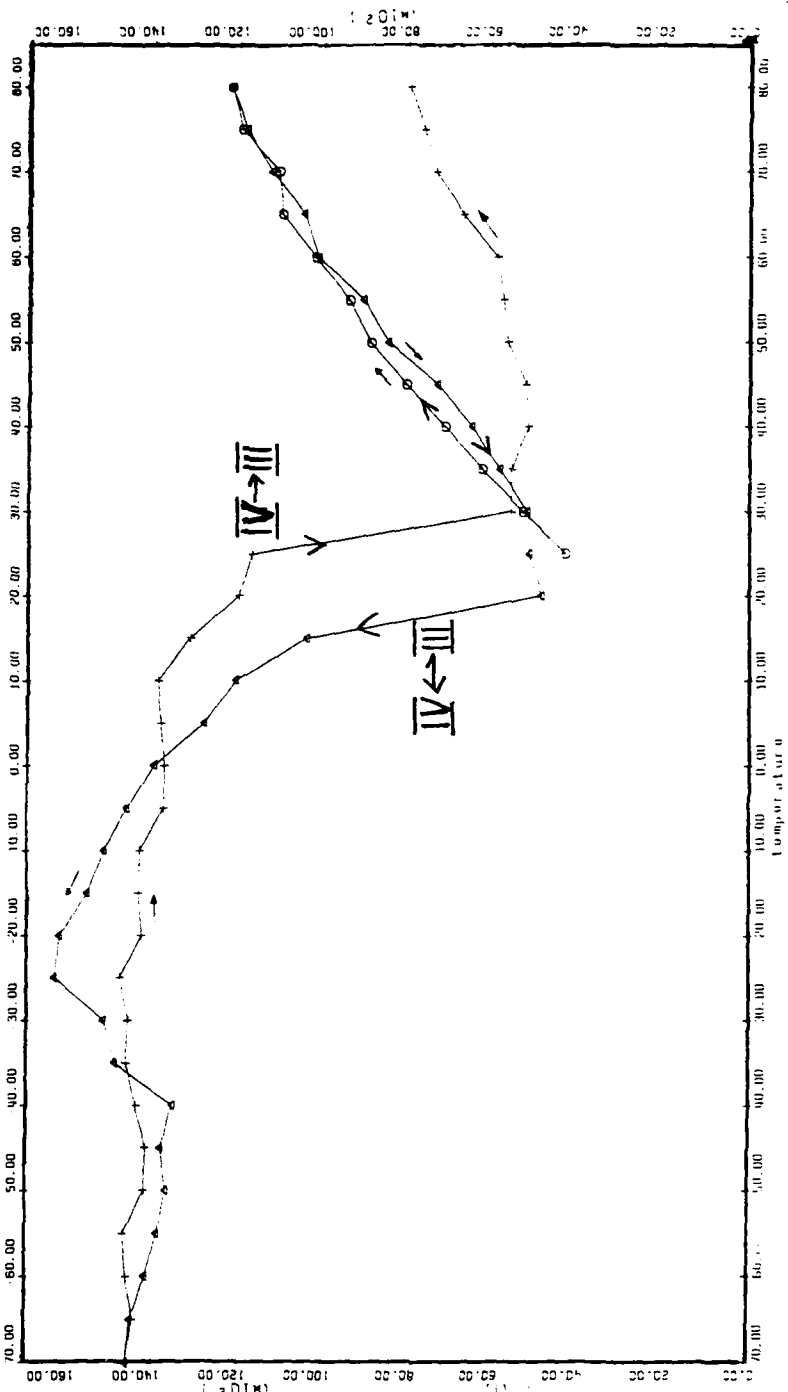
kf050988

20/80/-70/80°C



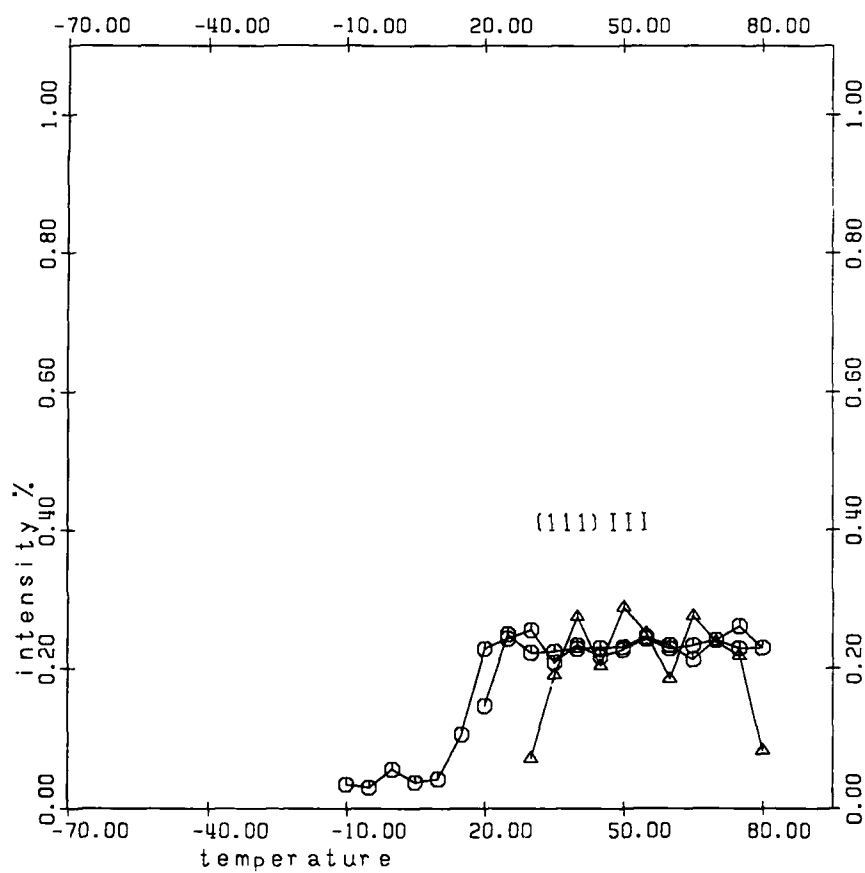
# Difference Curve Y(T)

kf050988  
20/80/-70/80°C



# Intensities

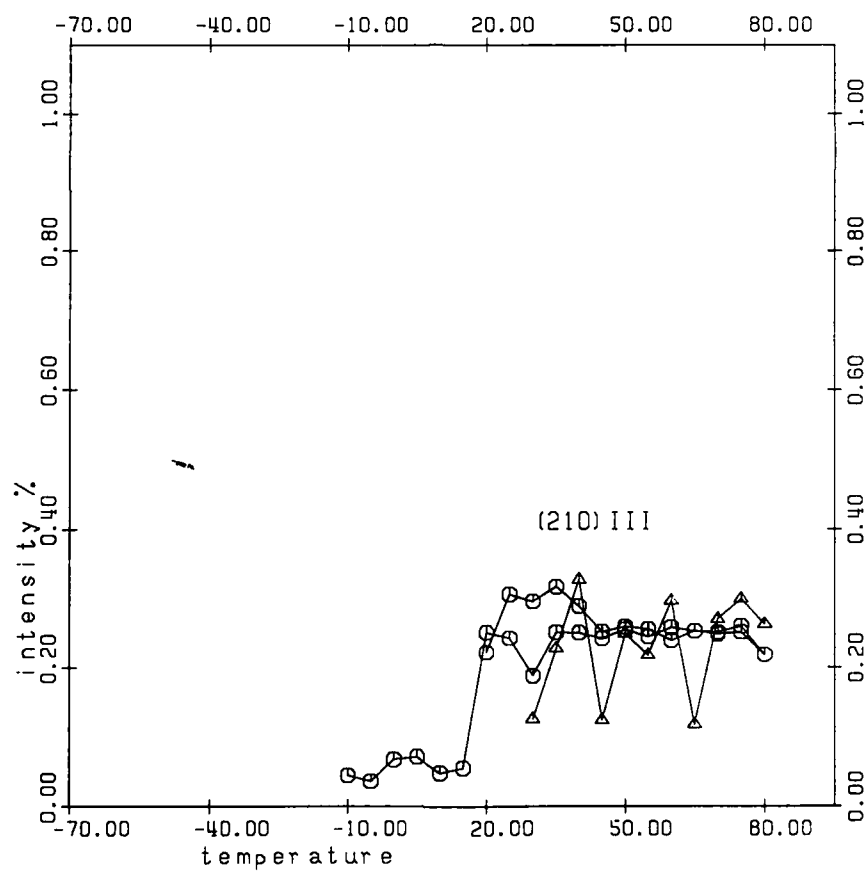
kf050988  
20/80/-70/80°C





# Intensities

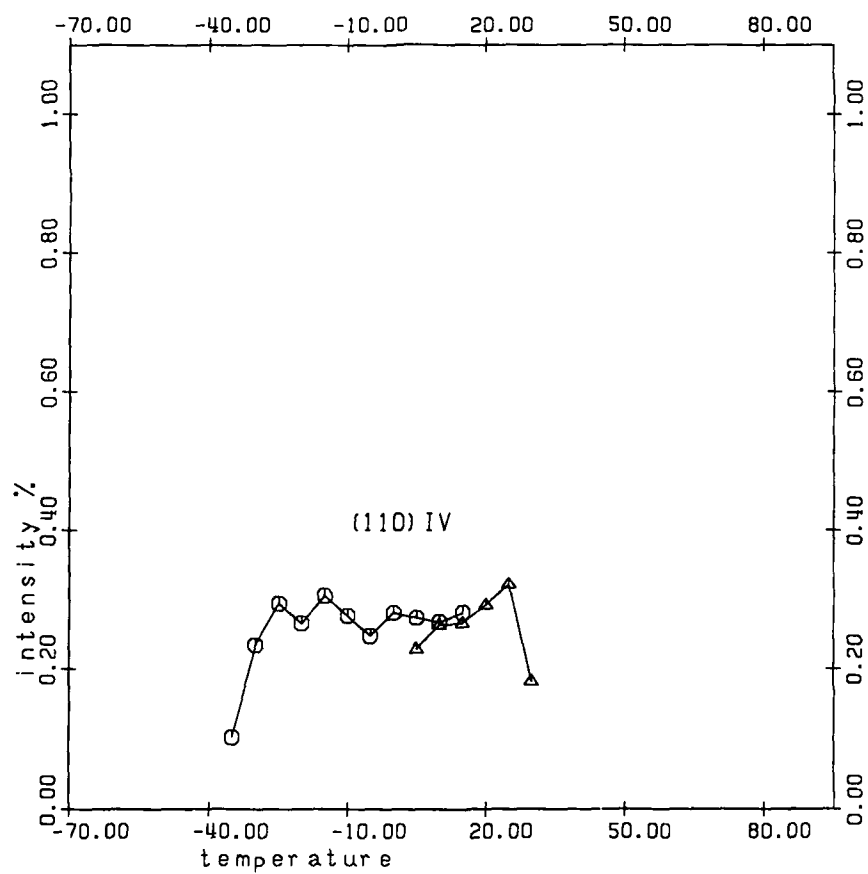
kf050988  
20/80/-70/80°C



### Intensities

kf050988

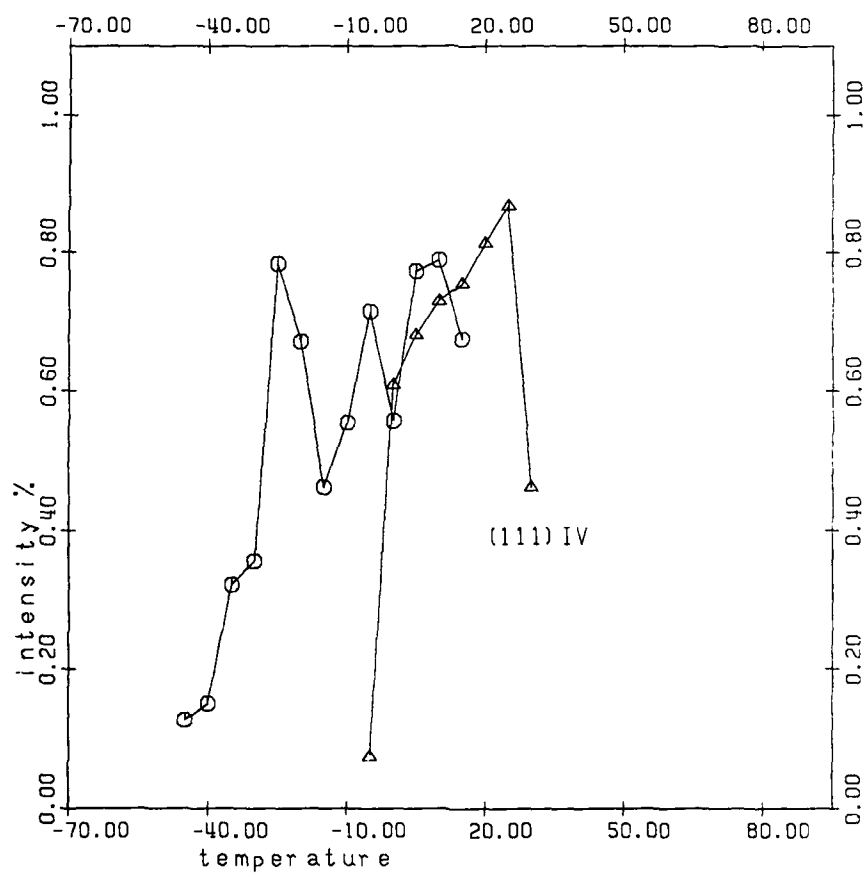
20/80/-70/80°C



# Intensities

kf050988

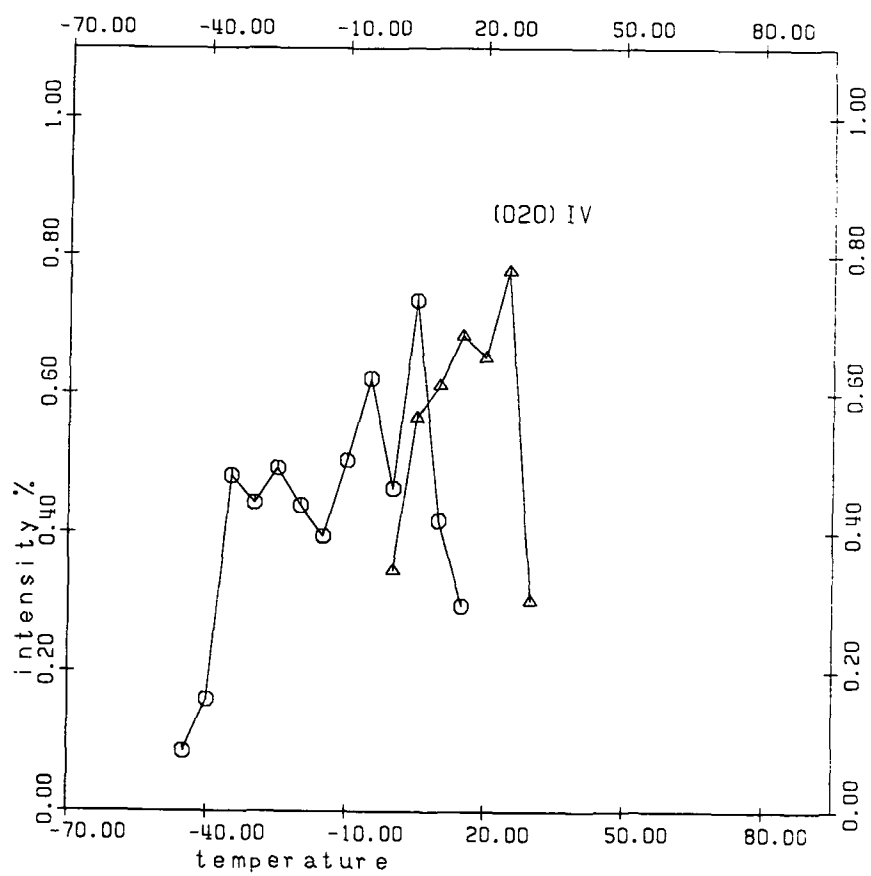
20/80/-70/80°C



# Intensities

kf050988

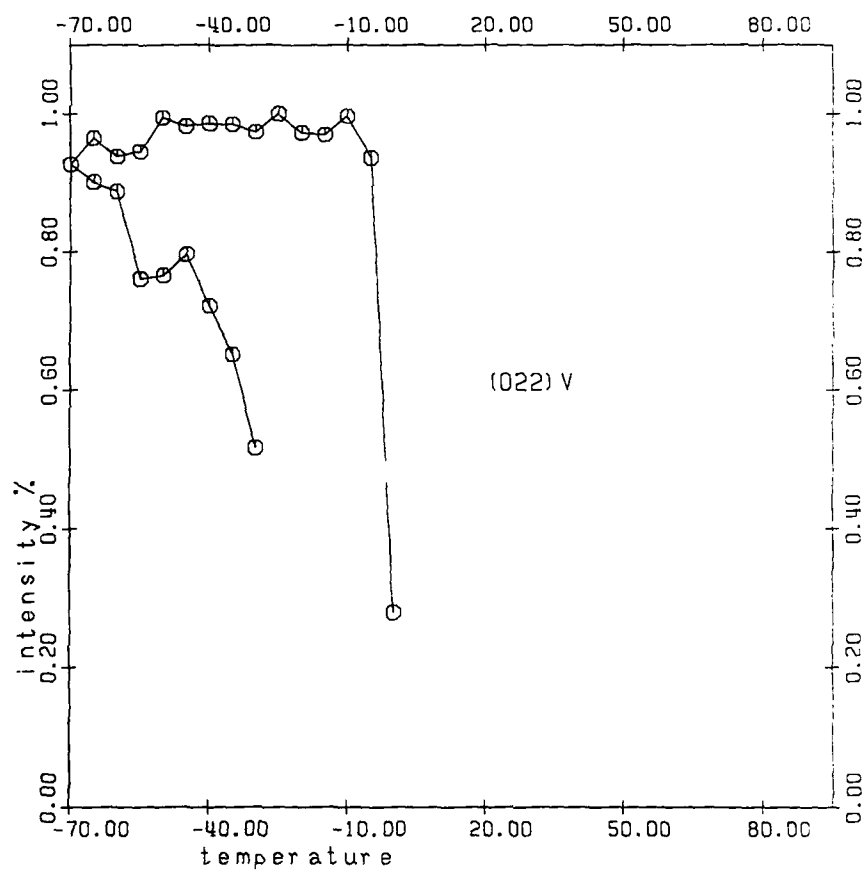
20/80/-70/80°C



# Intensities

kf050988

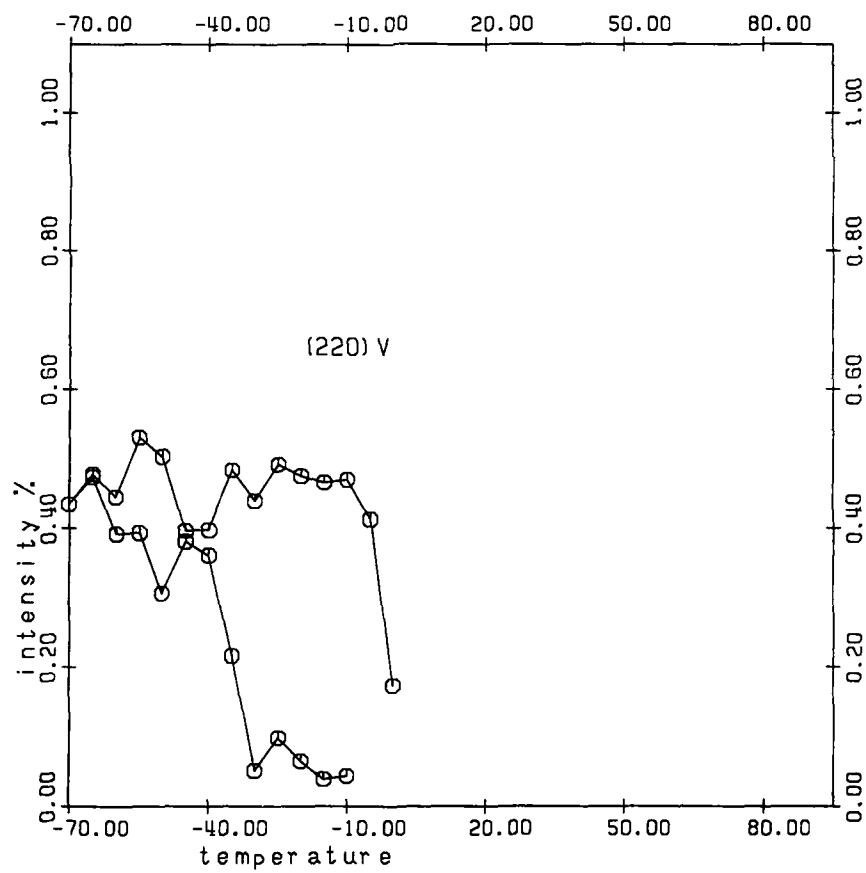
20/80/-70/80°C



# Intensities

kf050988

20/80/-70/80°C

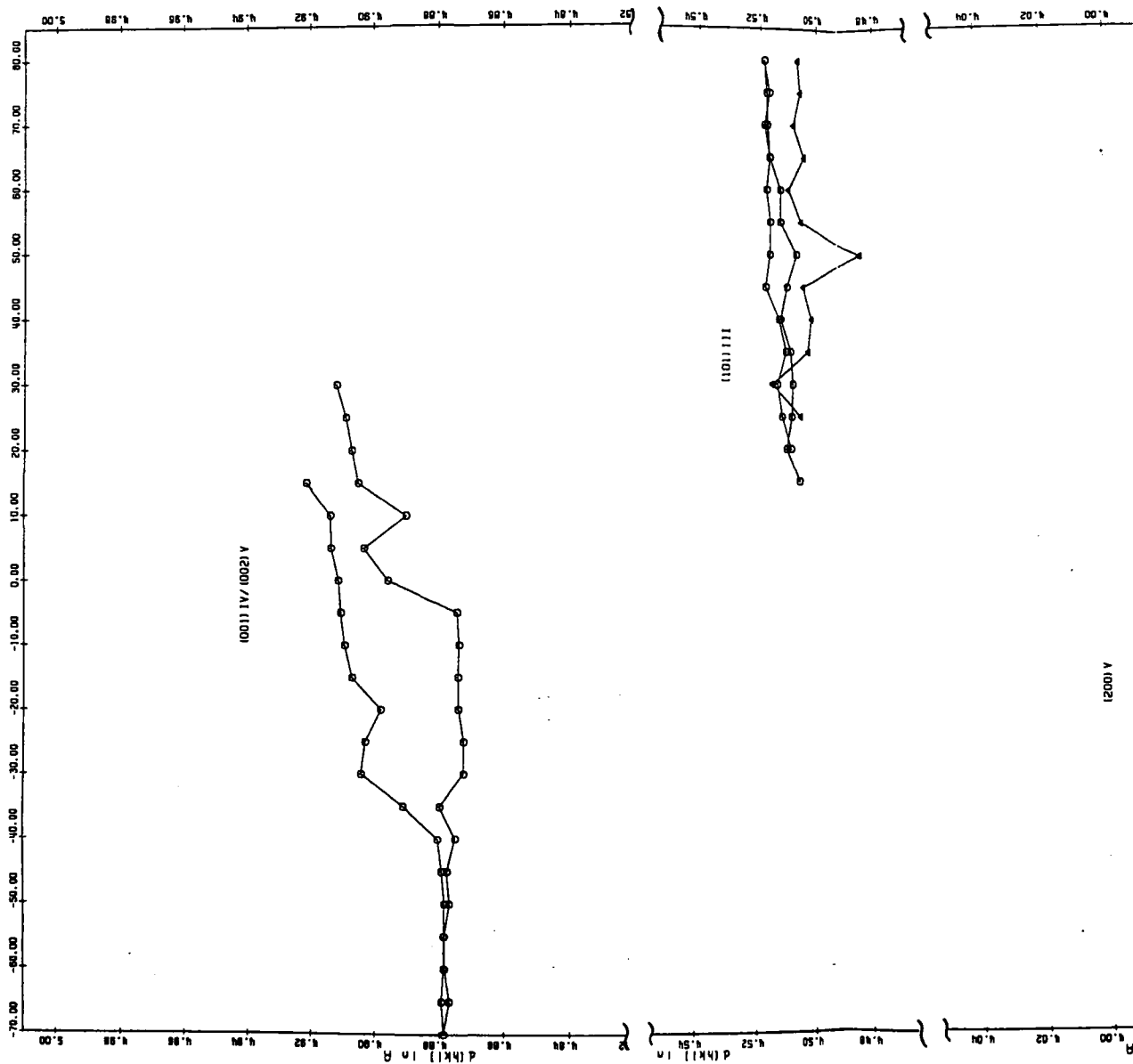


# Lattice Plane Distances

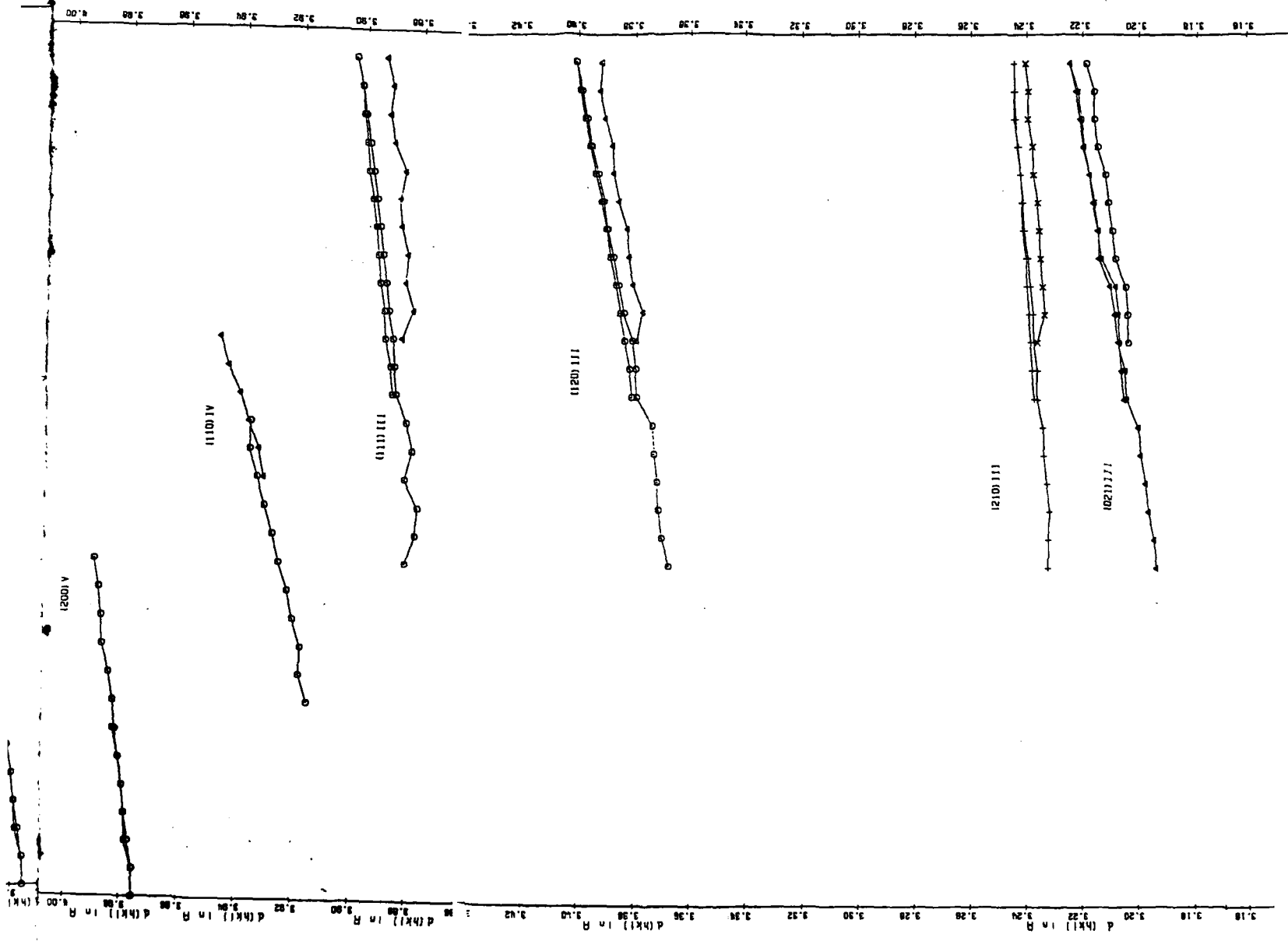
kf050988

20/80/-70/80°C

0 kf050988.dal 26 -83 1 \*



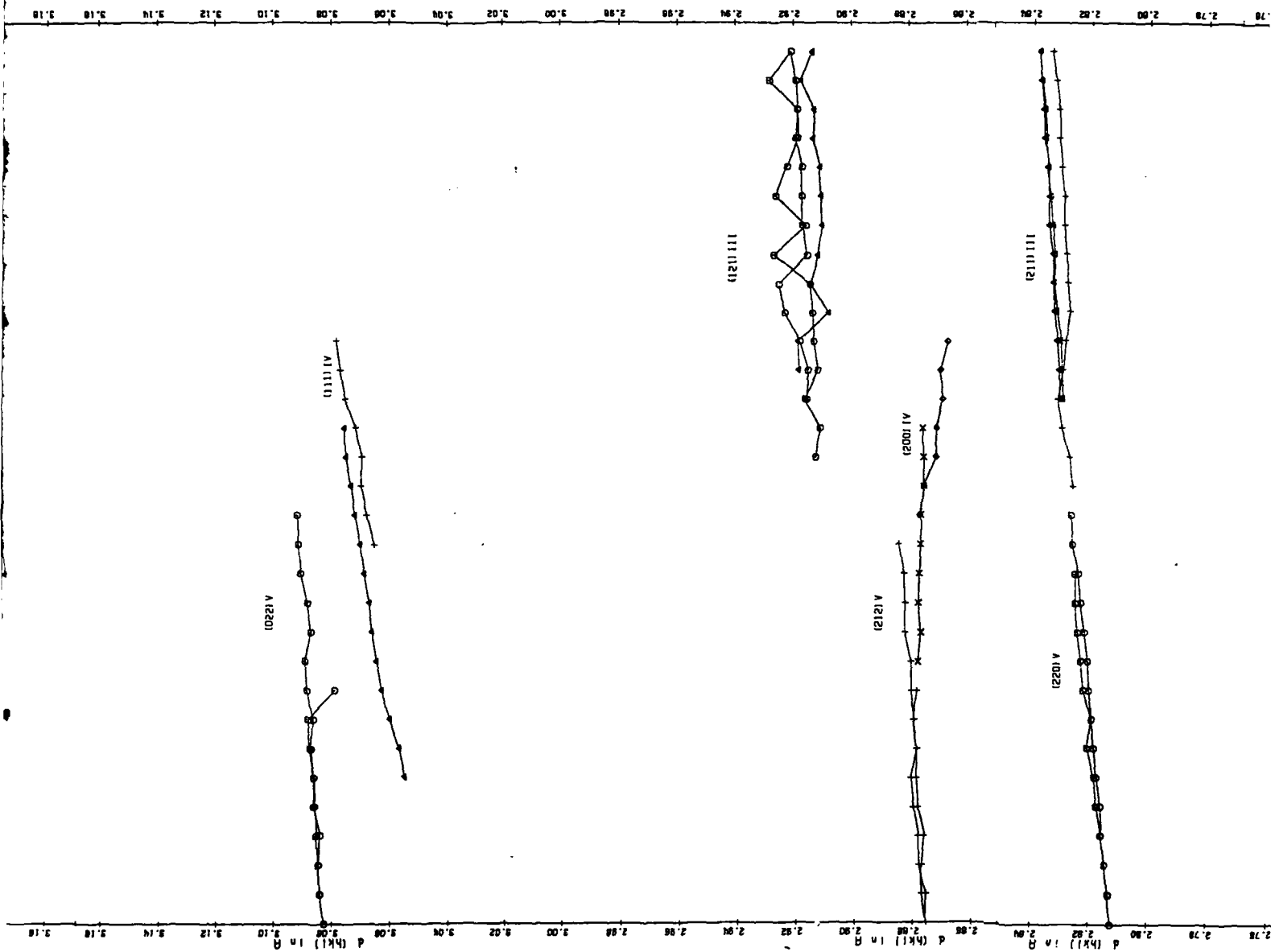
1075



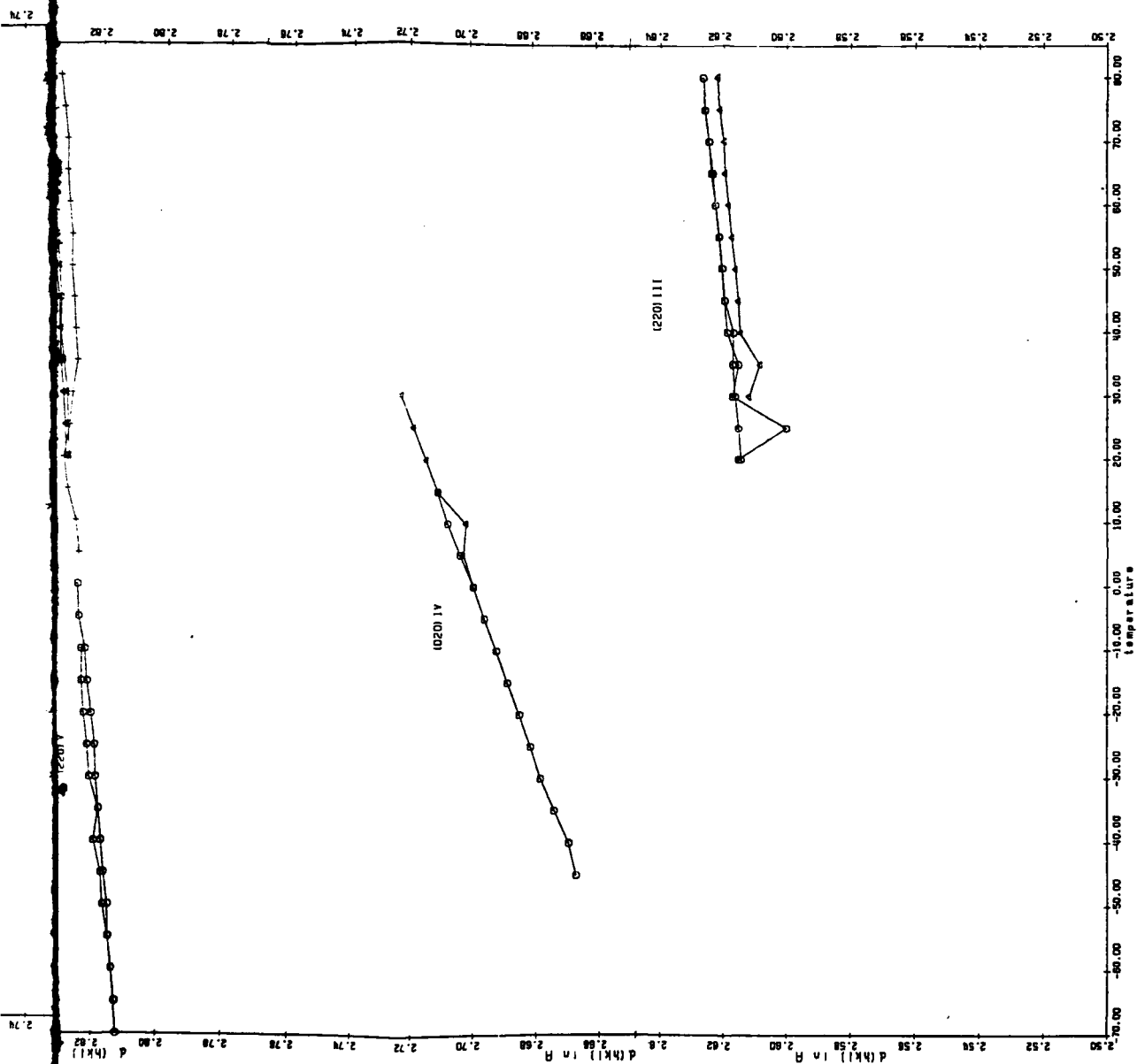
275

375





4 of 5

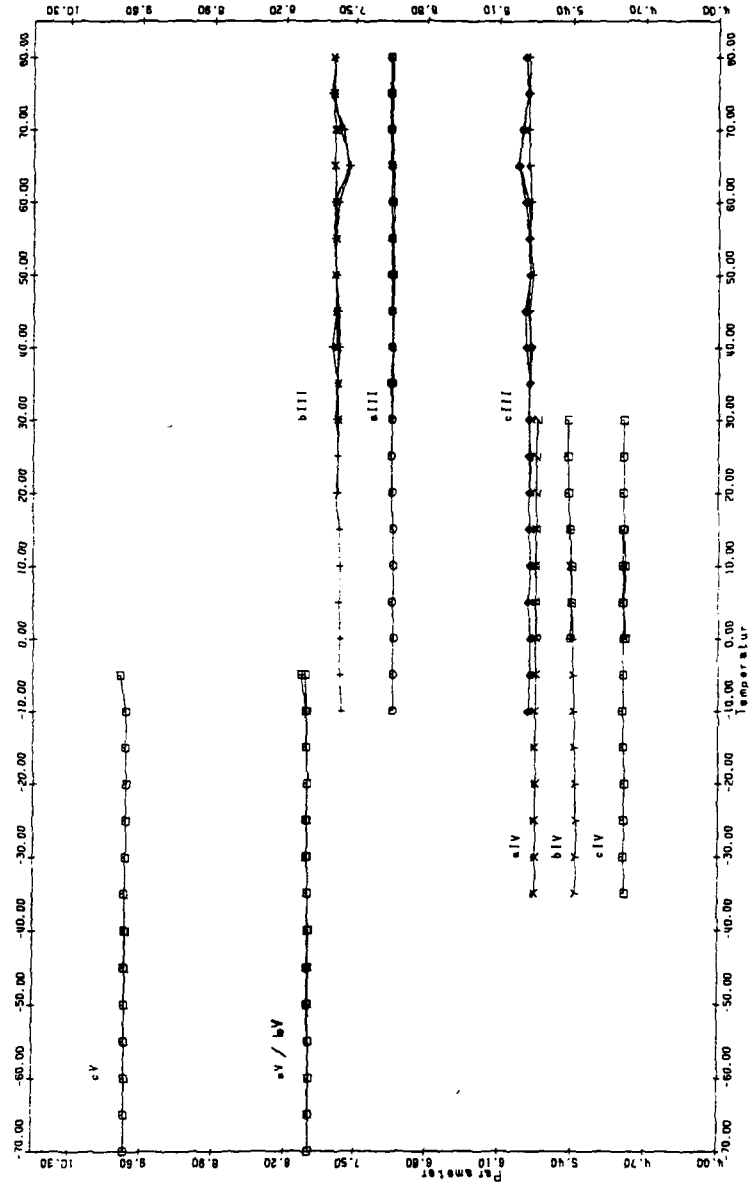


595

# Lattice Parameters

kf050988

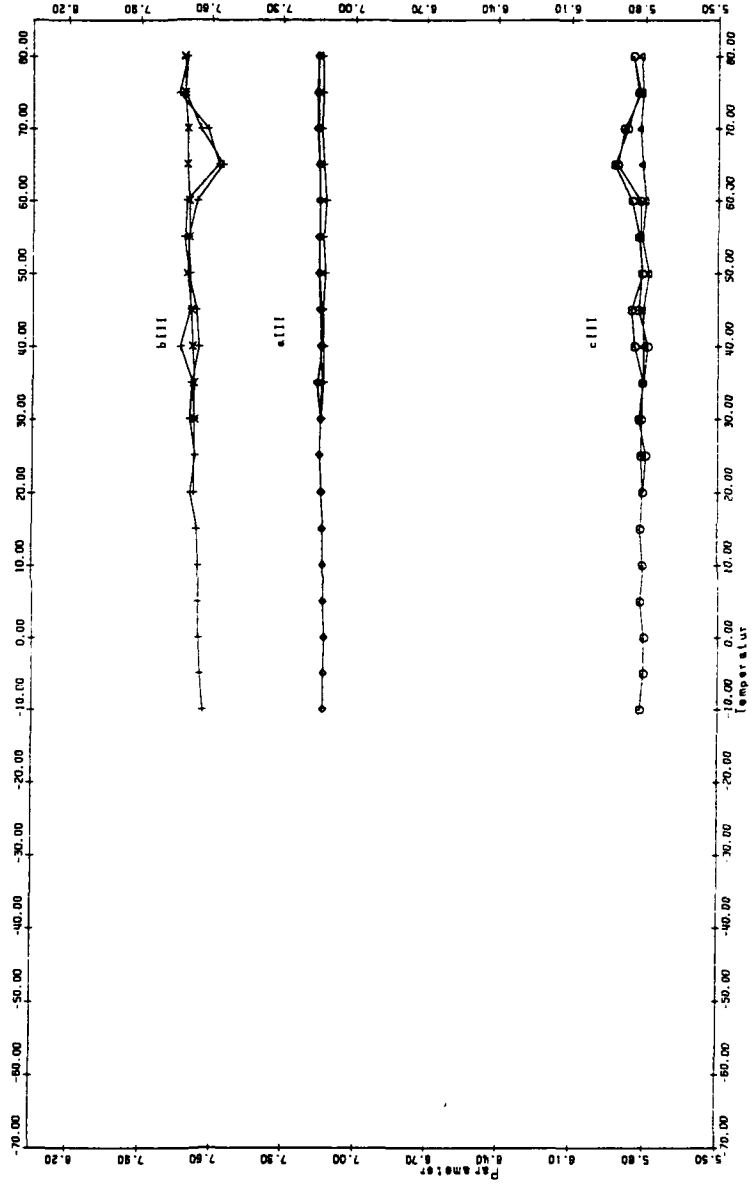
20/80/-70/20



# Lattice Parameters

kf050988

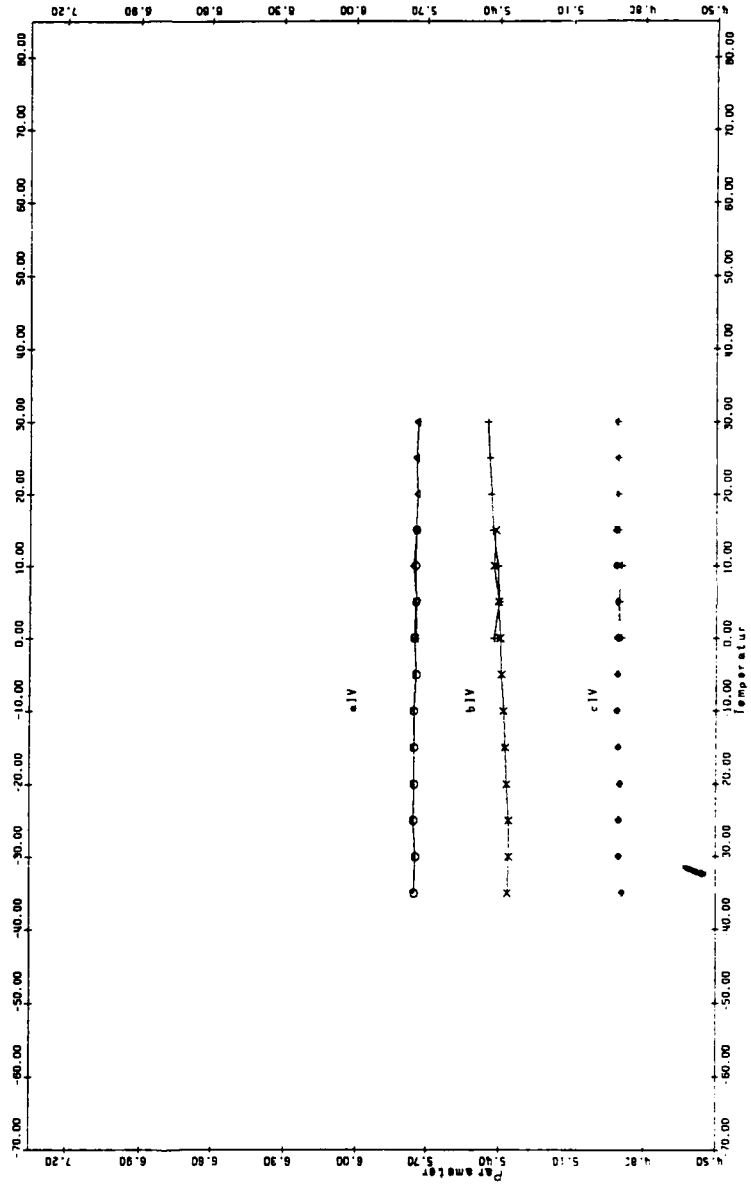
20/80/-70/20



# Lattice Parameters

kf050988

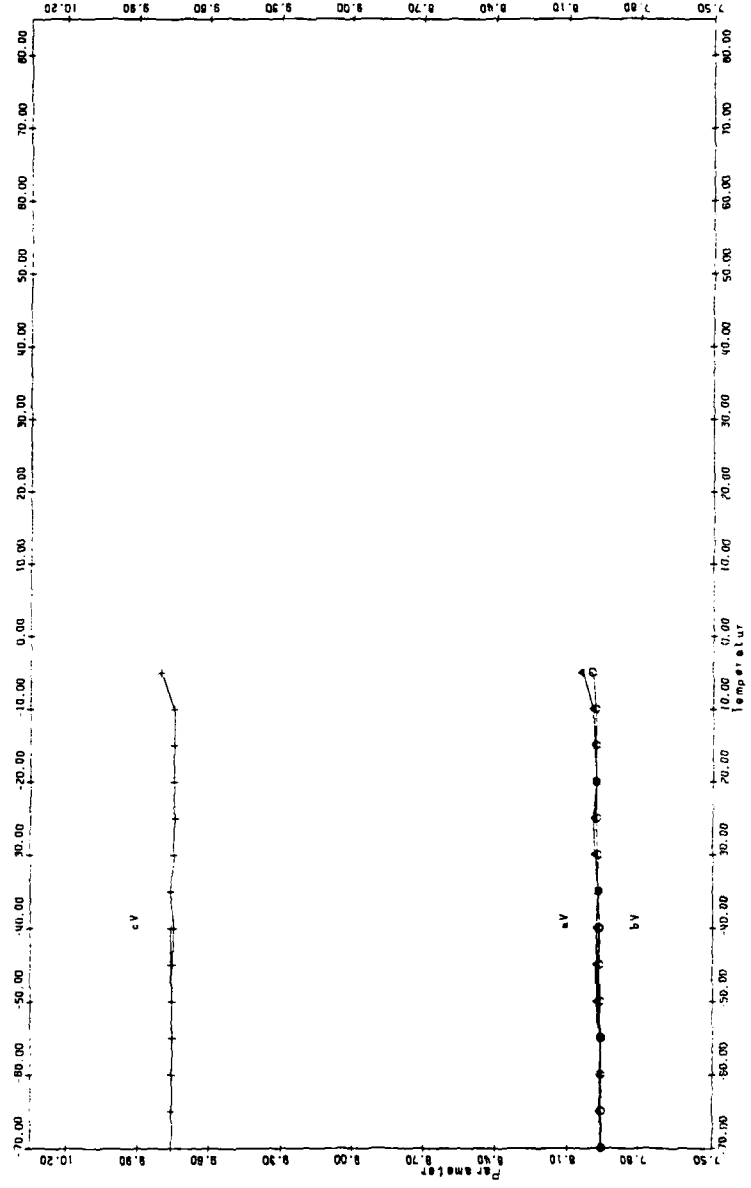
20/80/-70/20



# Lattice Parameters

kf050988

20/80/-70/20



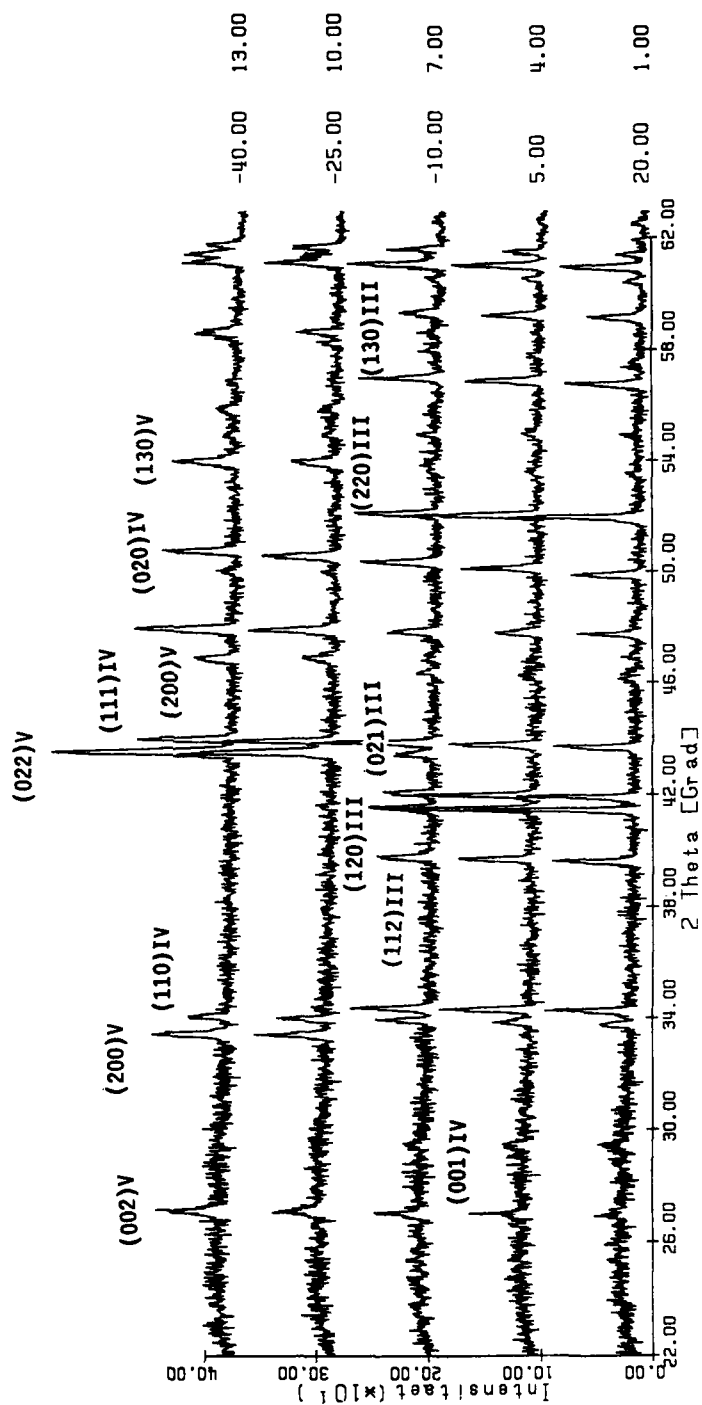
***Series***  
***KF 060988***

***Temperature***  
***Program***  
***20/—70/80/—70***

# Diffraction Patterns

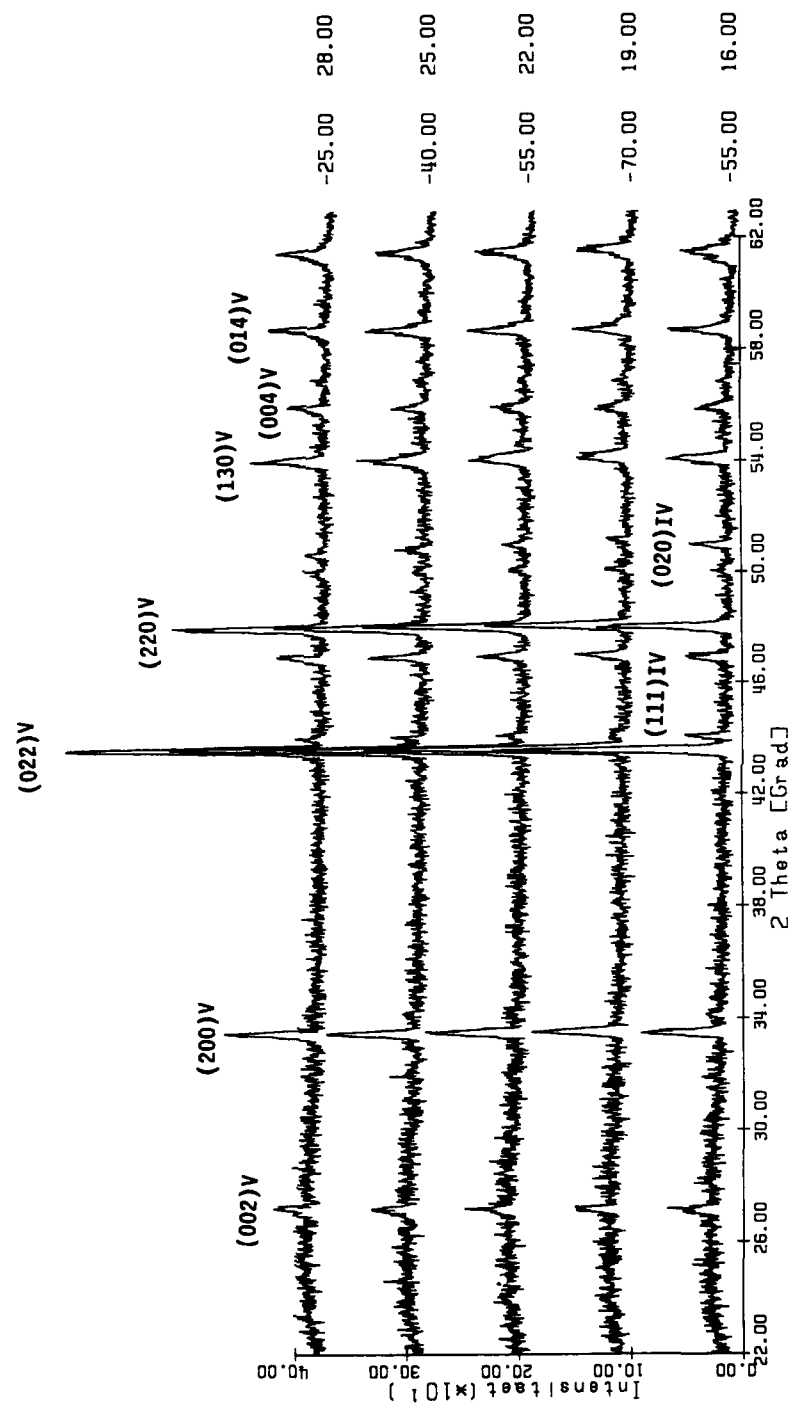
kf060988

20/-70/80/-70°C

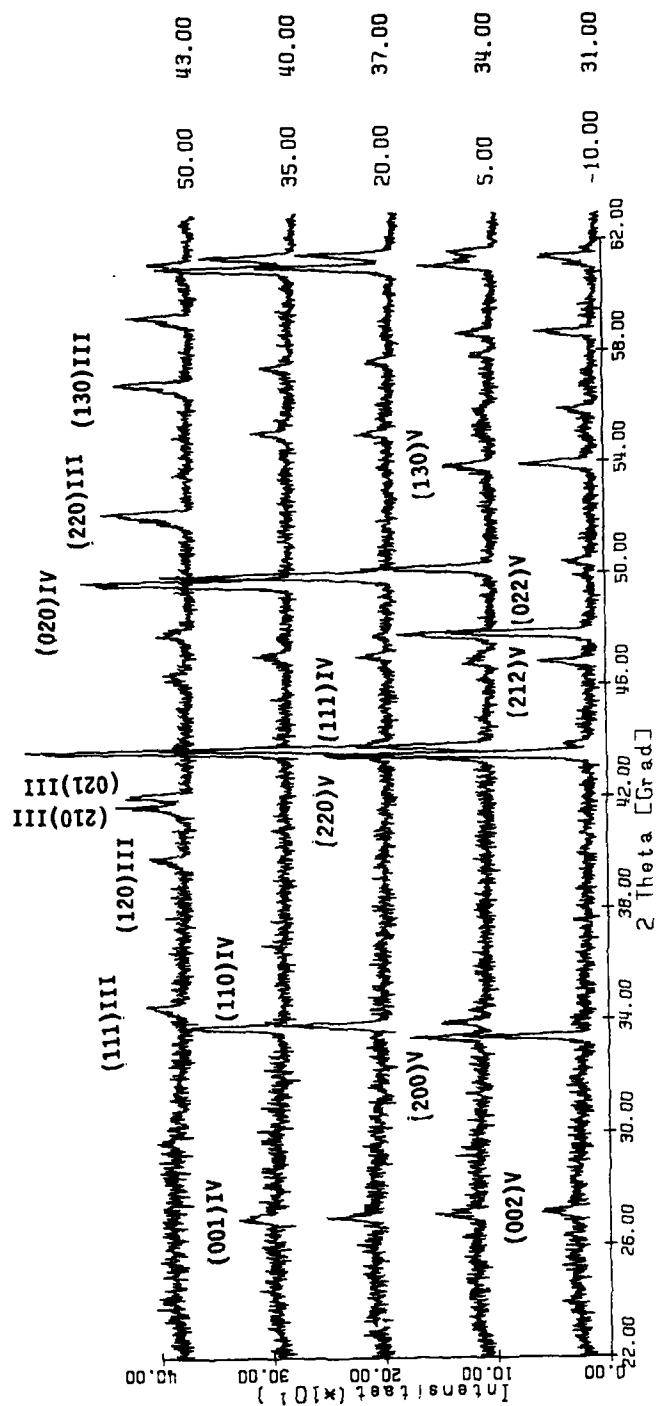




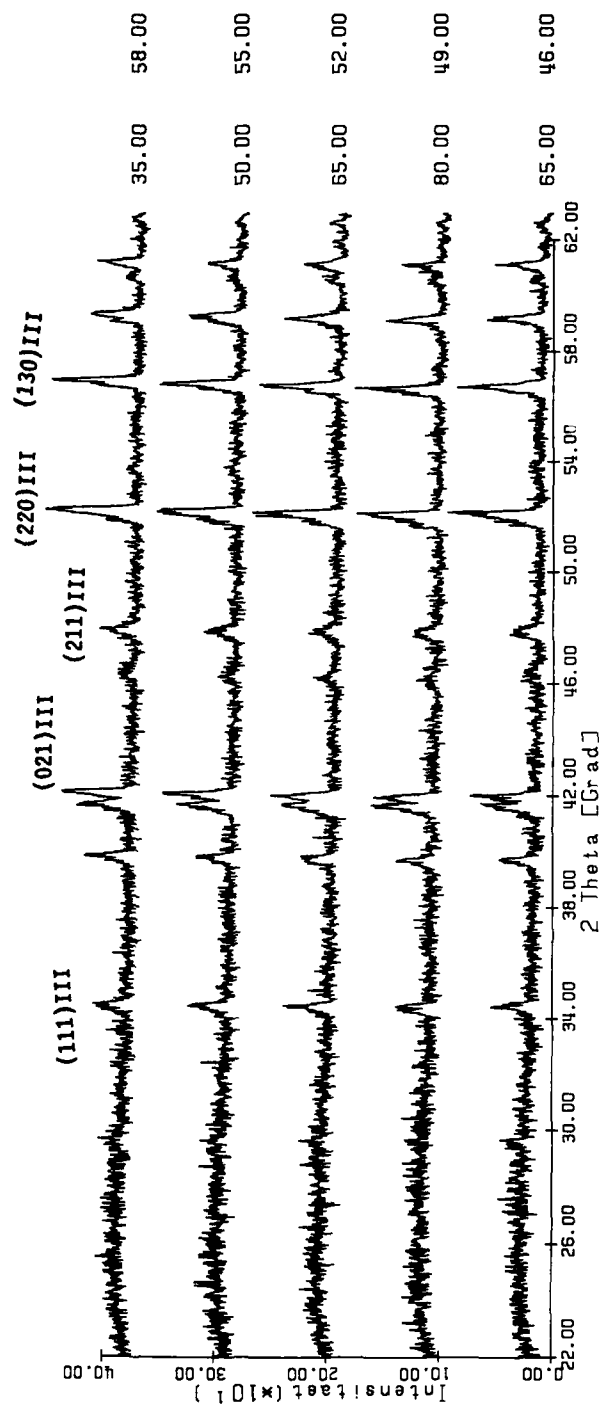
k f 060988



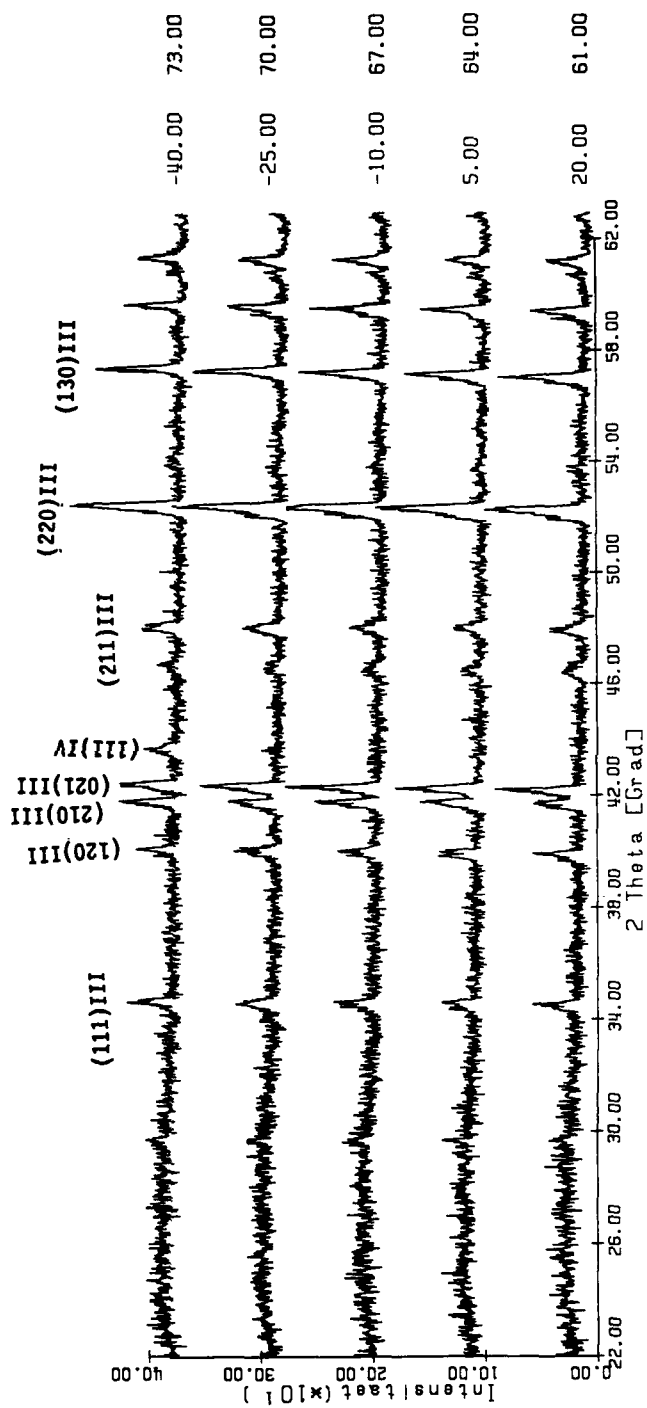
kf060988



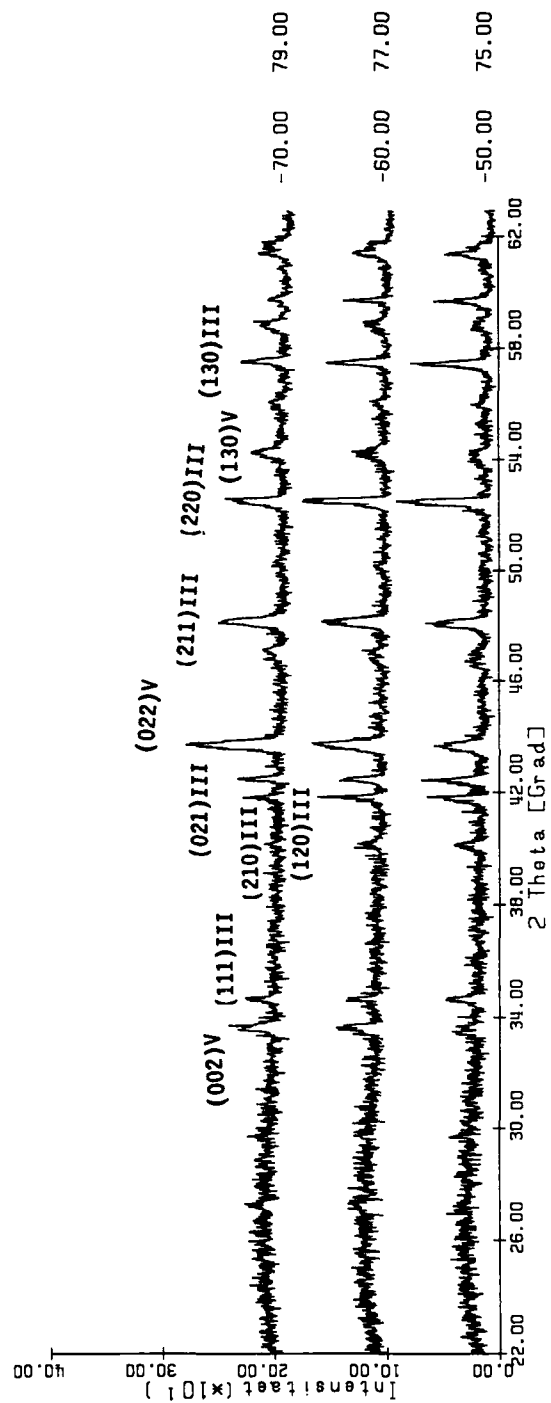
kf060988



kf060988



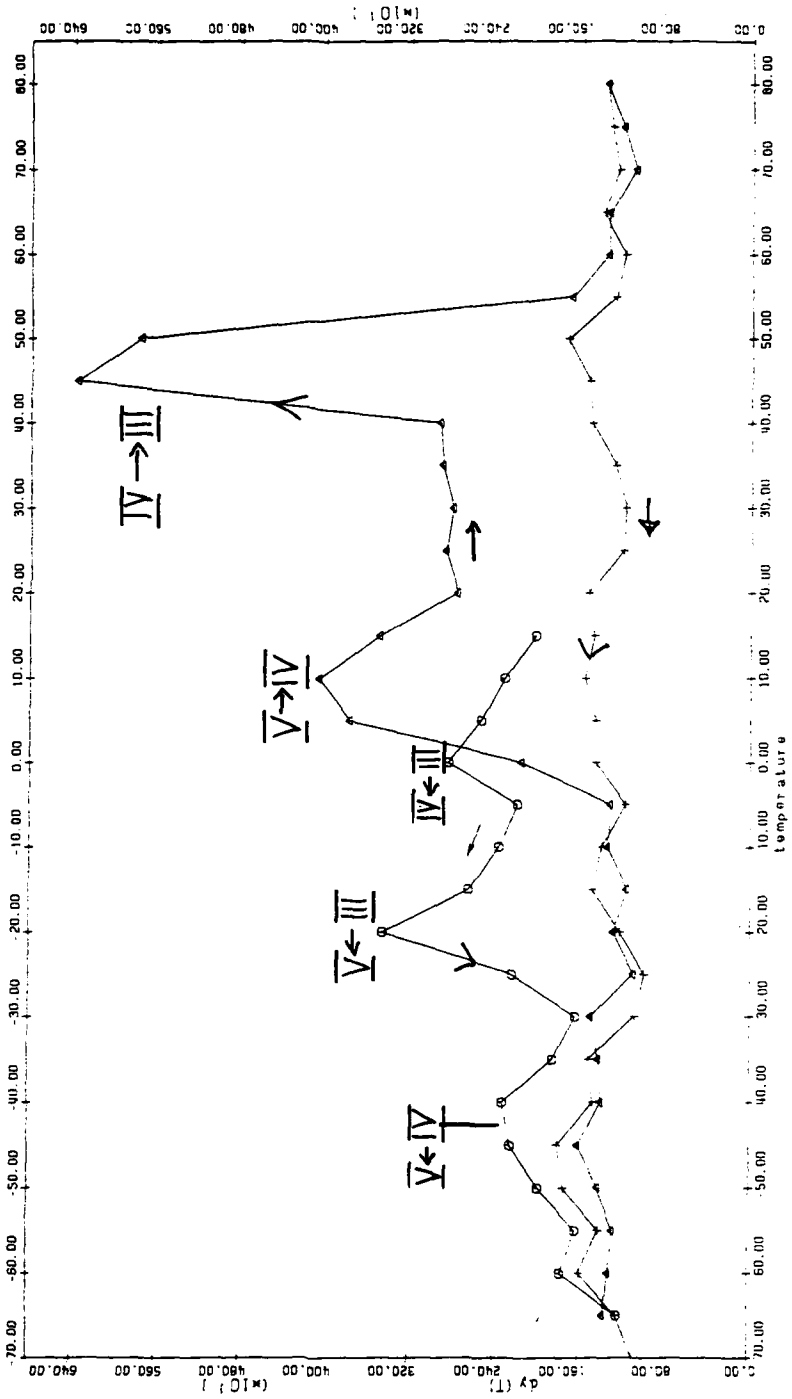
kf060988



# Difference Curve DY(T)

kf060988

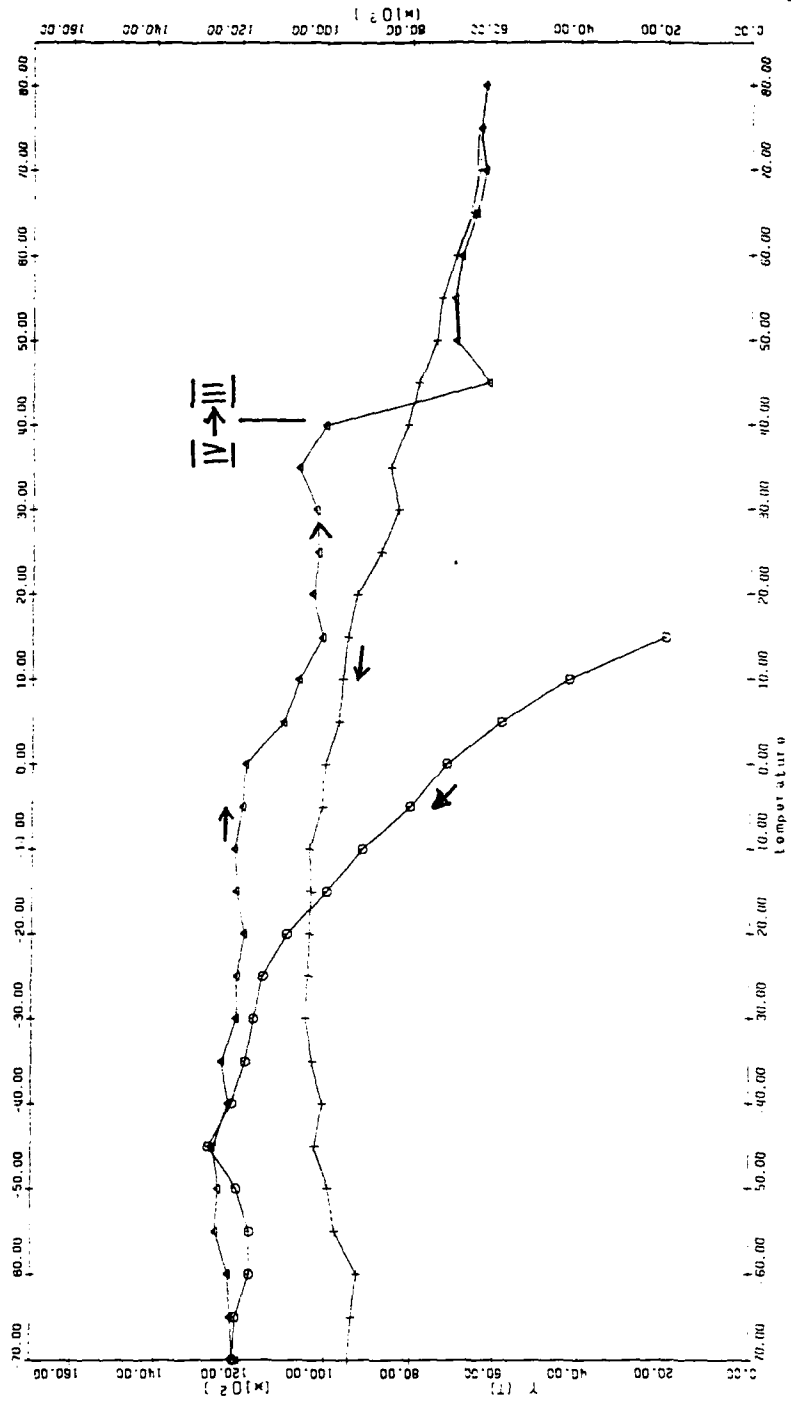
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# Difference Curve Y(T)

kf060988

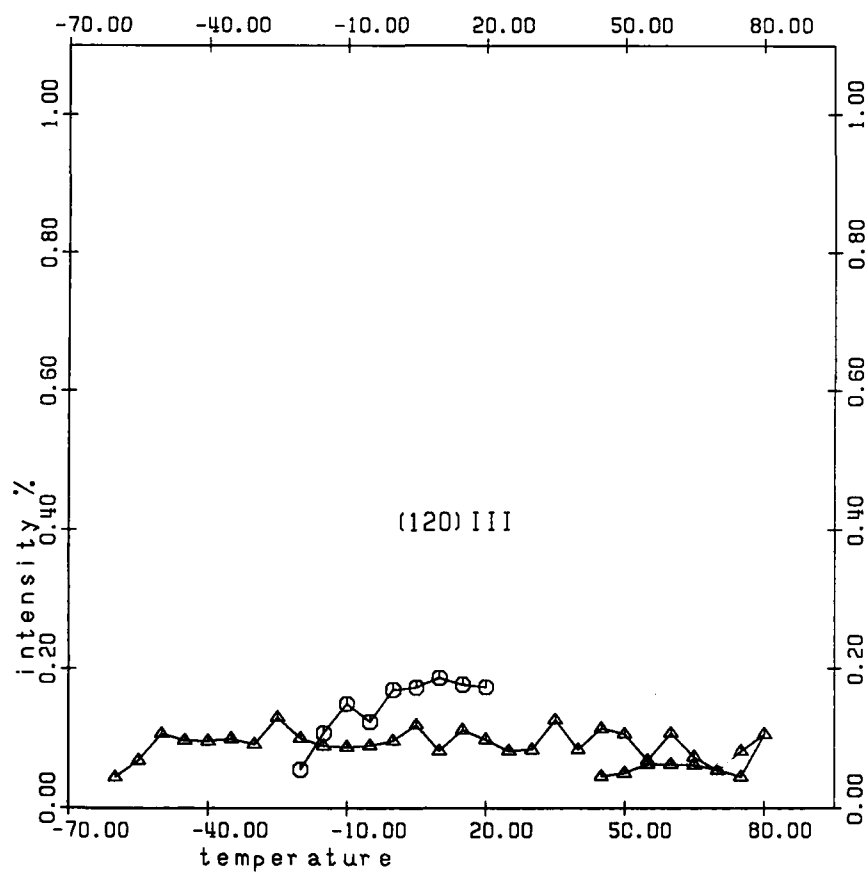
20/-70/80/-70°C



# Intensities

kf060988

20/-70/80/-70°C

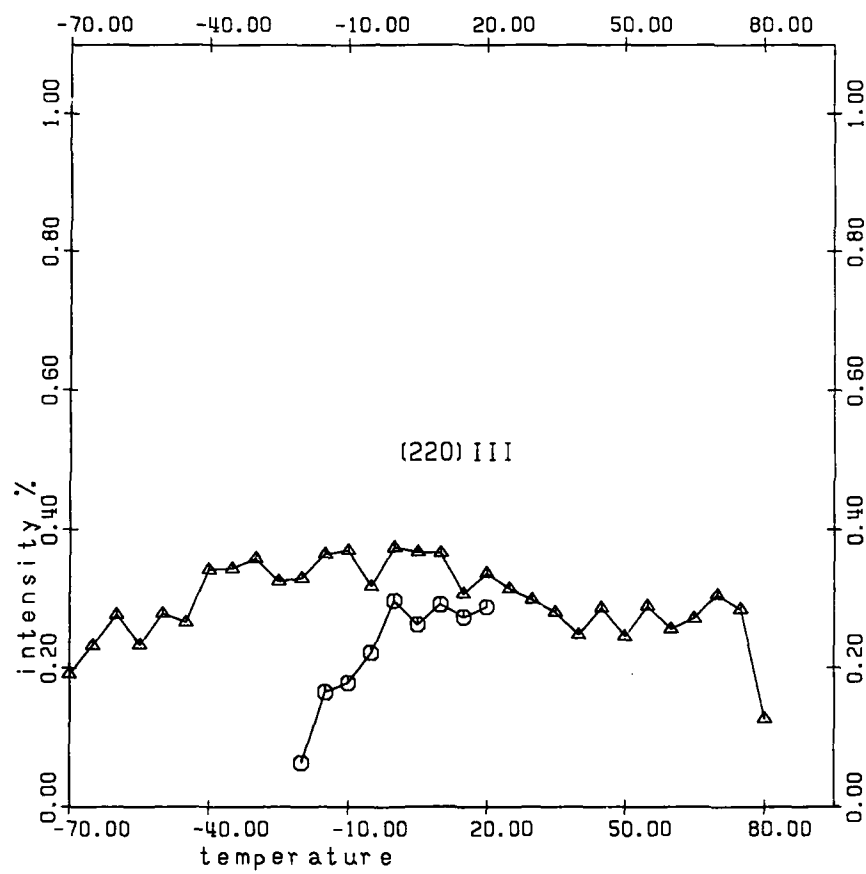




# Intensities

kf060988

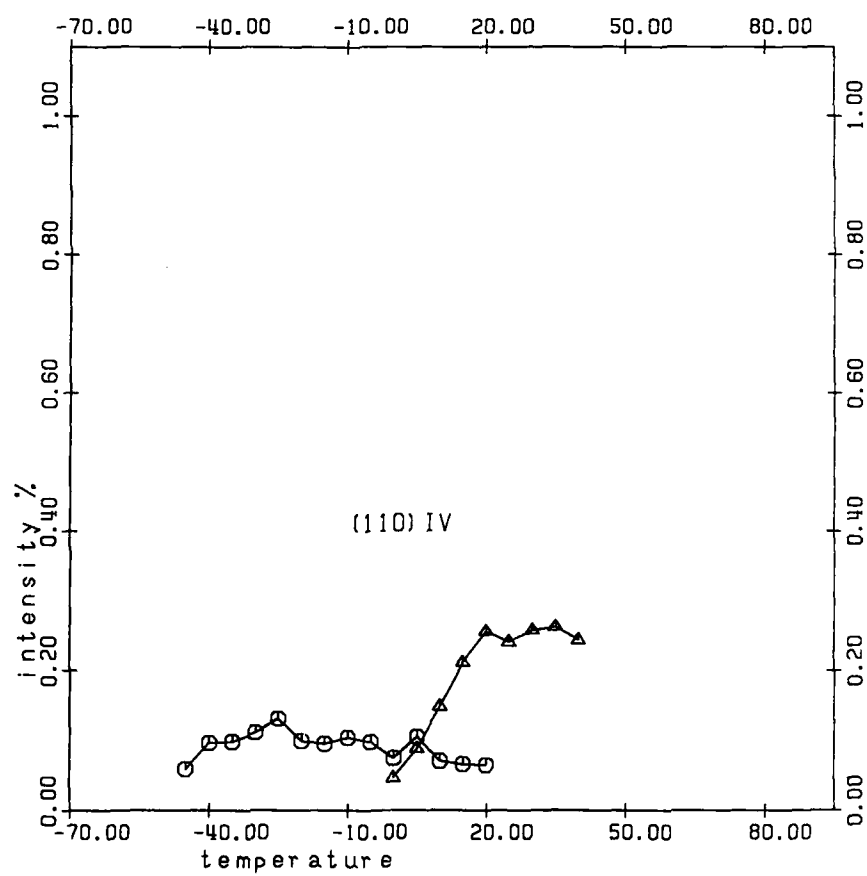
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# Intensities

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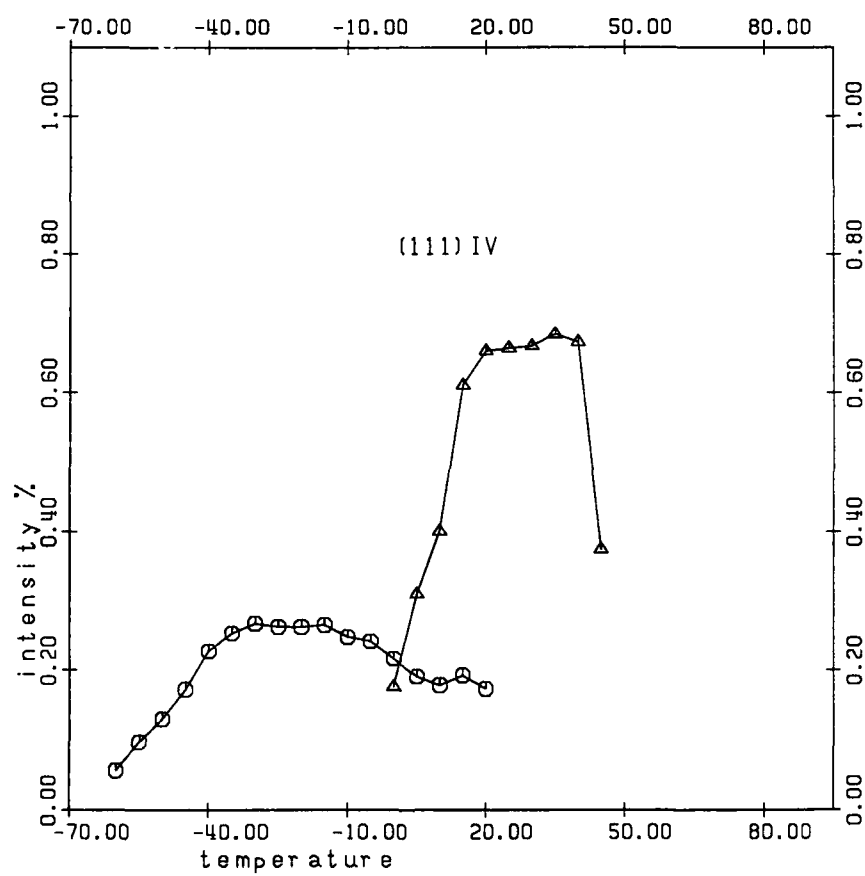
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# Intensities

kf060988

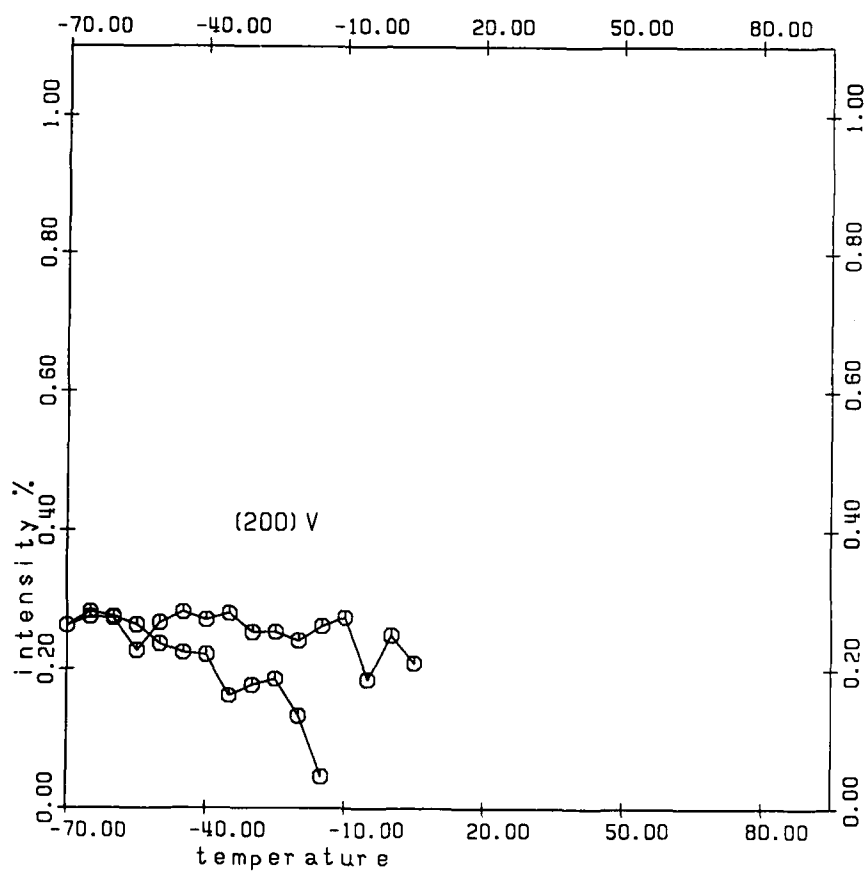
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# Intensities

kf060988

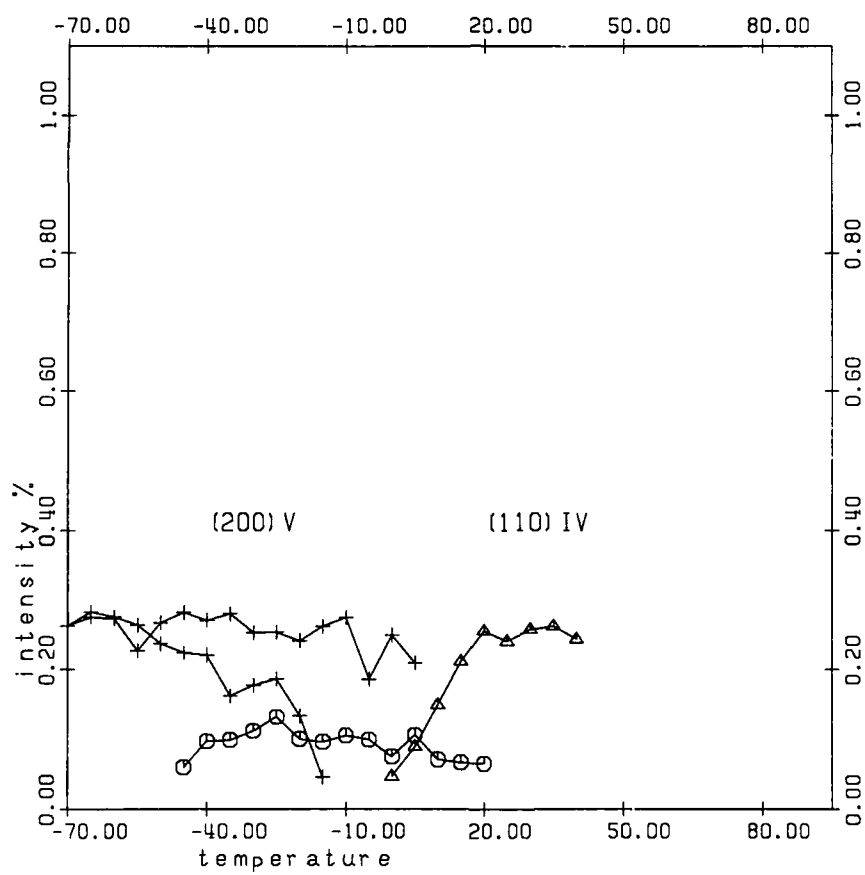
20/-70/80/-70°C



## Intensities

kf060988

20/-70/80/-70°C

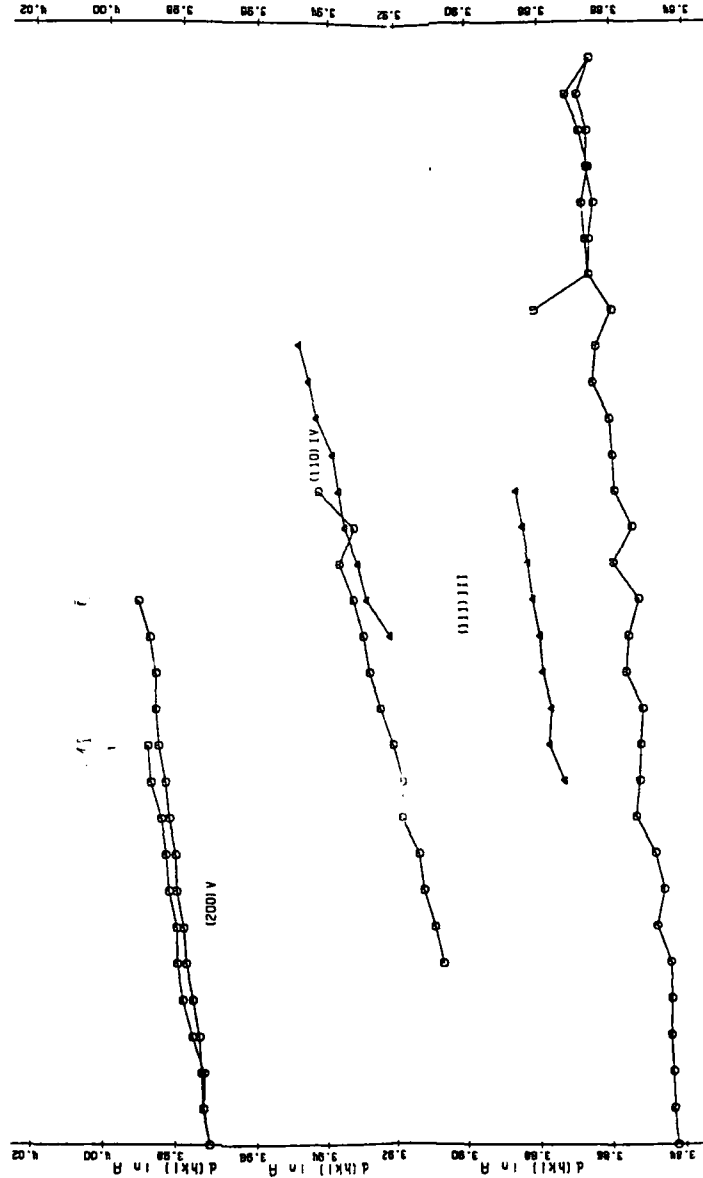
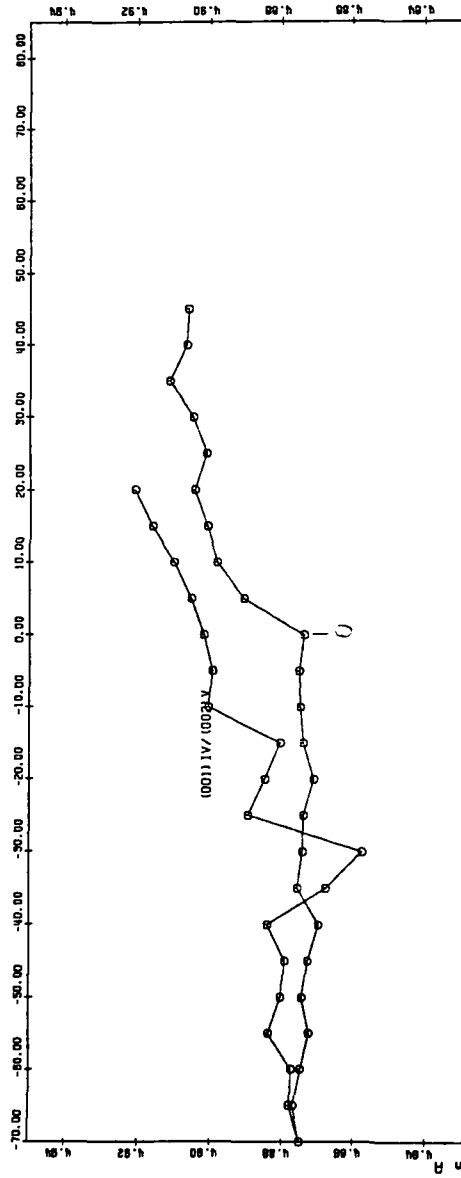


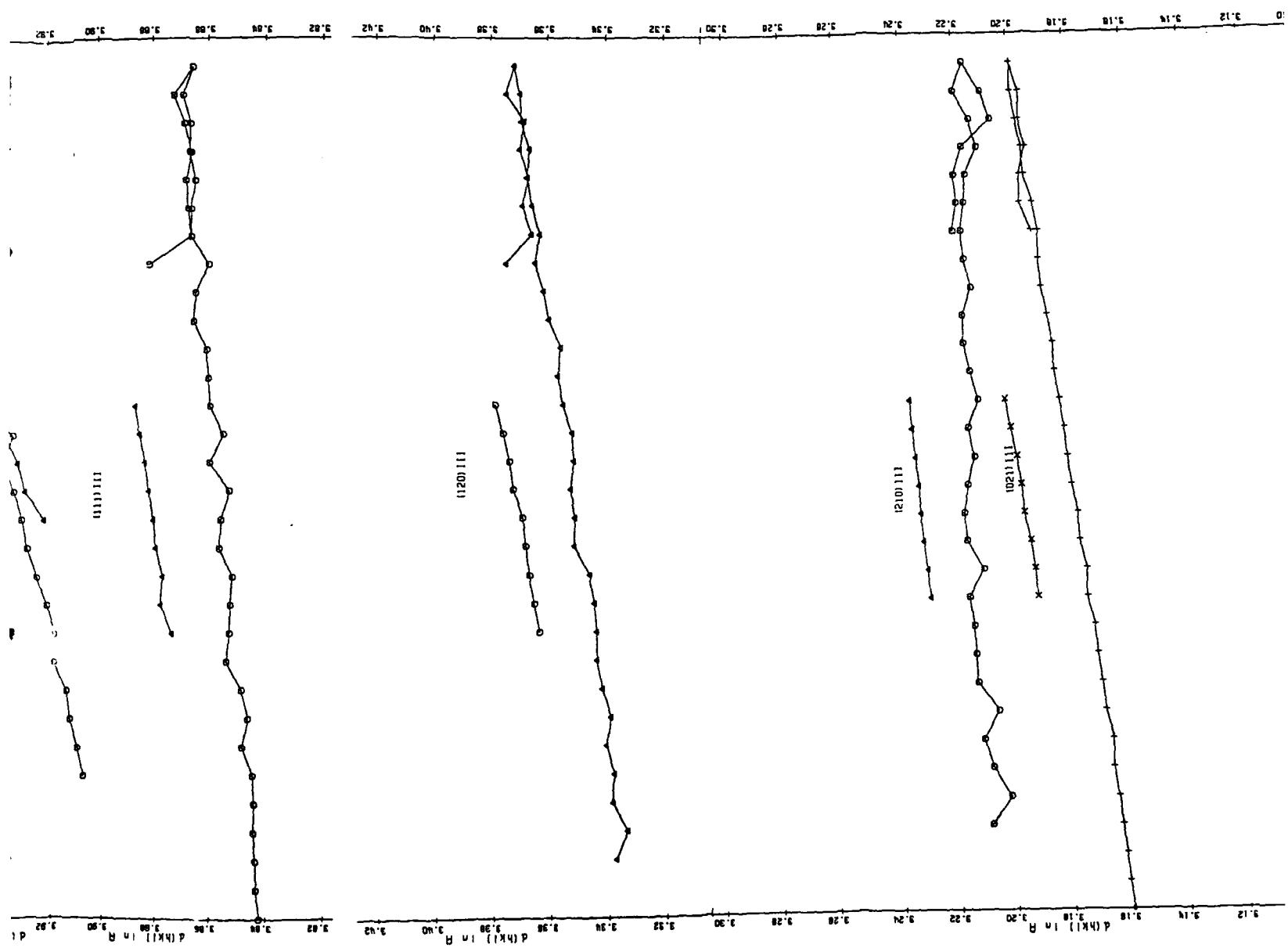
# Lattice Plane Distances

kf060988

20/-70/80/-70°C

Q kf060988.dat 1 -42 1





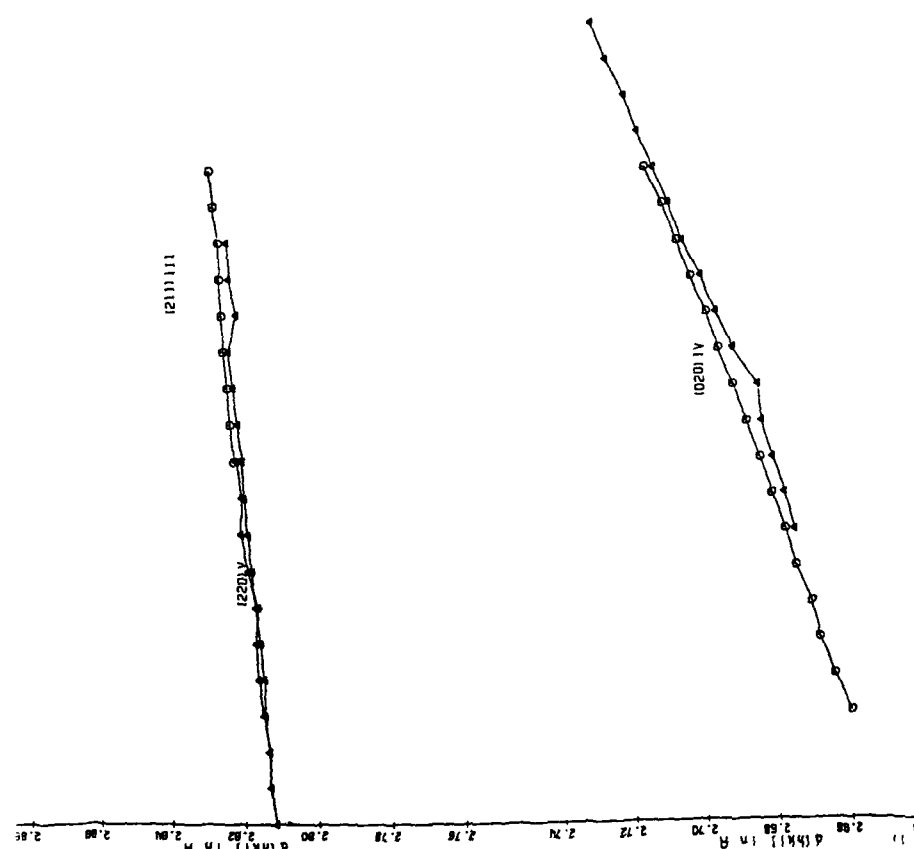
2085

30

2.68 2.66 2.64 2.62 2.60 2.58 2.56 2.54 2.52 2.50 2.48 2.46 2.44 2.42 2.40 2.38 2.36 2.34 2.32 2.30 2.28 2.26 2.24 2.22 2.20 2.18 2.16 2.14 2.12 2.10 2.08 2.06 2.04 2.02 2.00 1.98 1.96 1.94 1.92 1.90 1.88 1.86 1.84 1.82 1.80 1.78 1.76 1.74 1.72 1.70 1.68 1.66 1.64 1.62 1.60 1.58 1.56 1.54 1.52 1.50 1.48 1.46 1.44 1.42 1.40 1.38 1.36 1.34 1.32 1.30 1.28 1.26 1.24 1.22 1.20 1.18 1.16 1.14 1.12 1.10 1.08 1.06 1.04 1.02 1.00 0.98 0.96 0.94 0.92 0.90 0.88 0.86 0.84 0.82 0.80 0.78 0.76 0.74 0.72 0.70 0.68 0.66 0.64 0.62 0.60 0.58 0.56 0.54 0.52 0.50 0.48 0.46 0.44 0.42 0.40 0.38 0.36 0.34 0.32 0.30 0.28 0.26 0.24 0.22 0.20 0.18 0.16 0.14 0.12 0.10 0.08 0.06 0.04 0.02 0.00

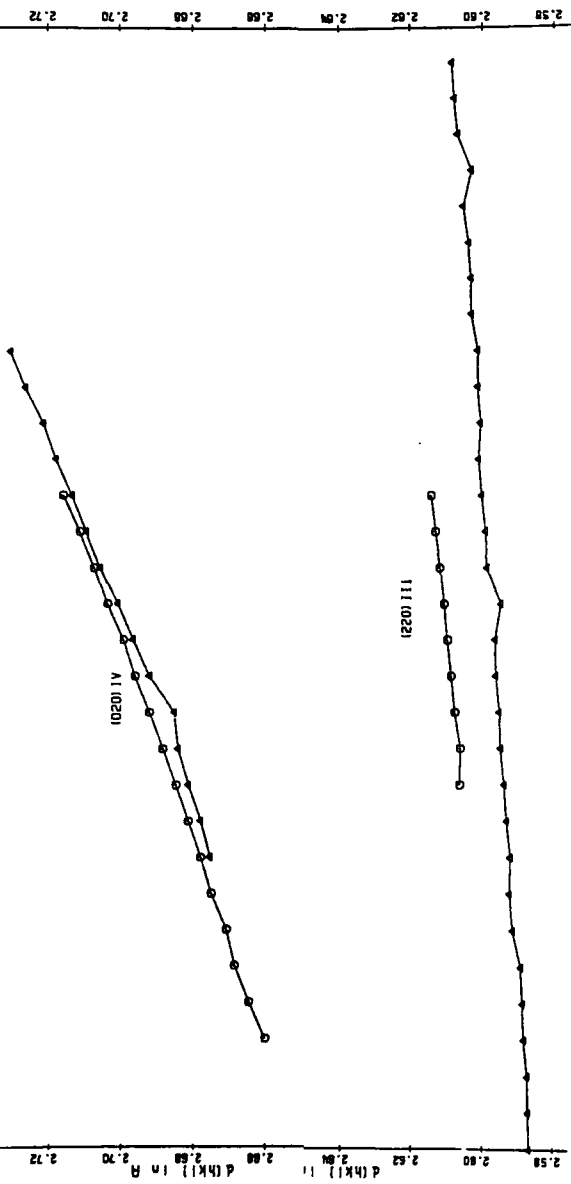
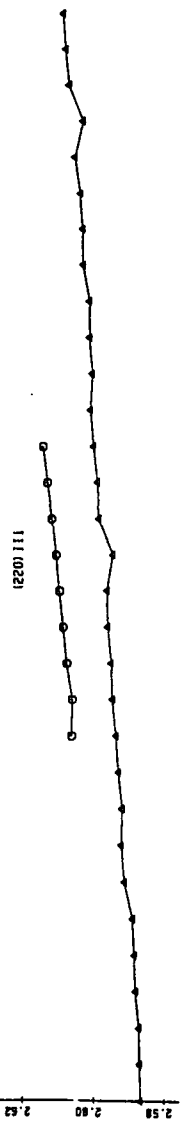
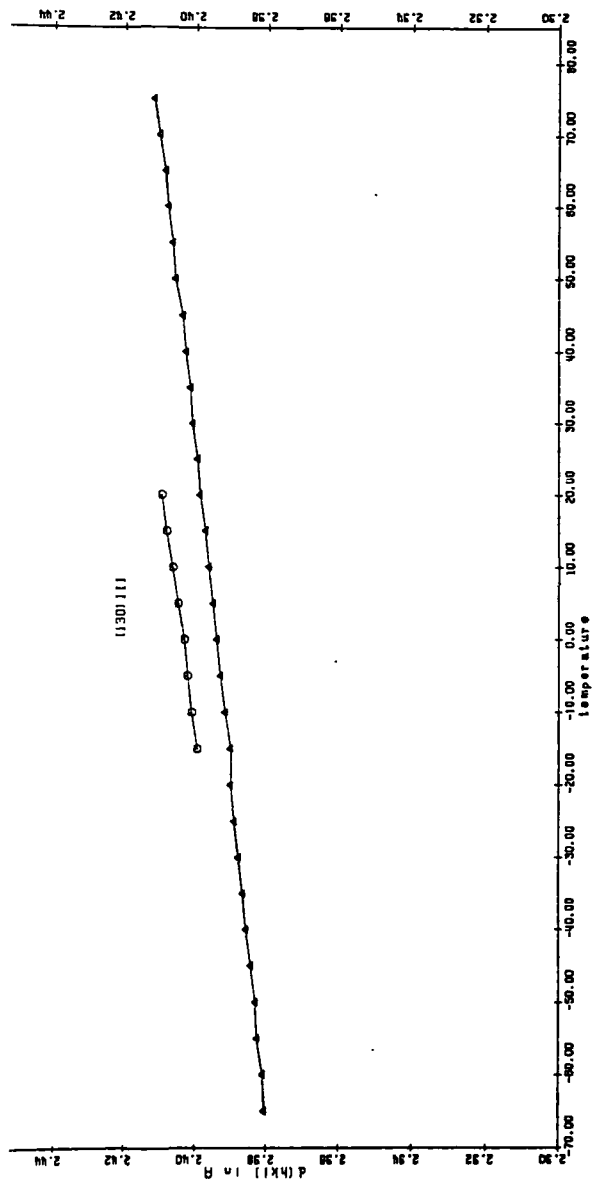


385



485



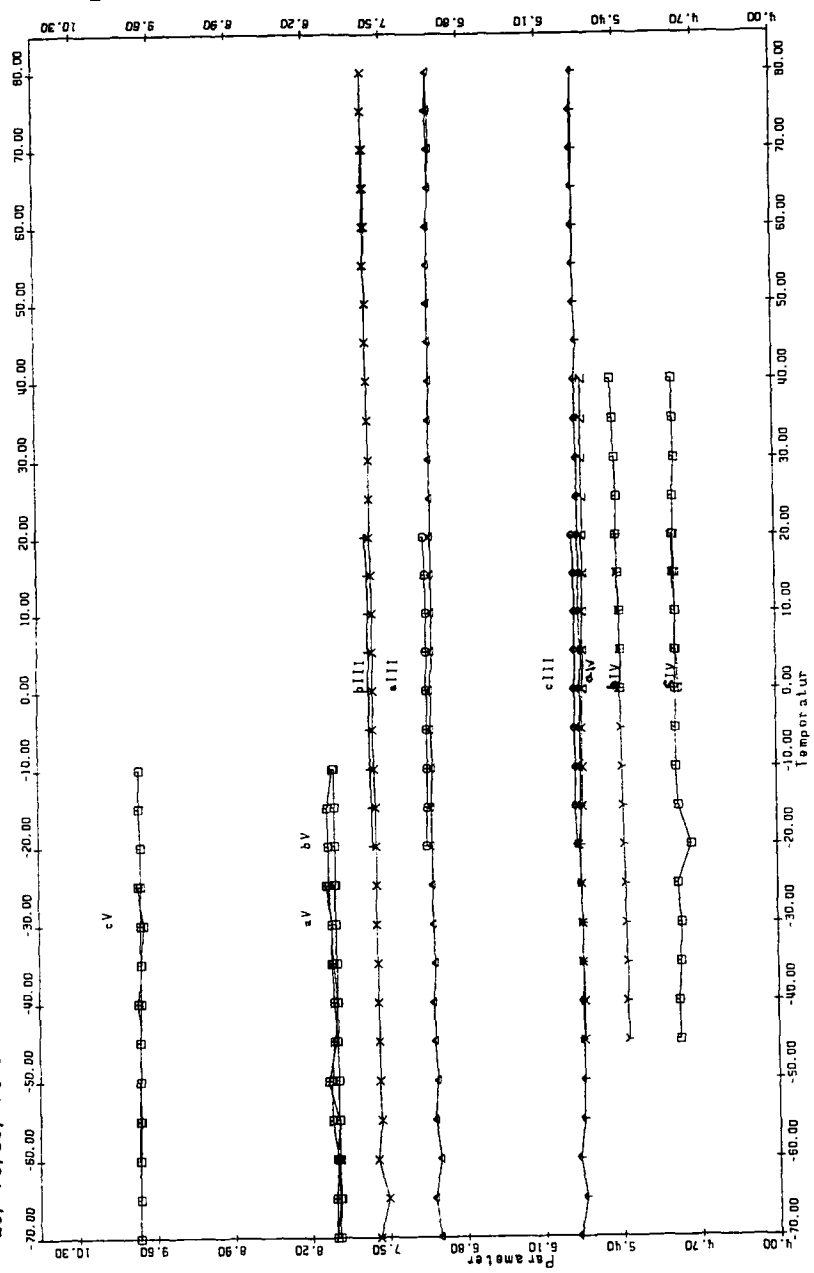


58'

# Lattice Parameters

kf060988

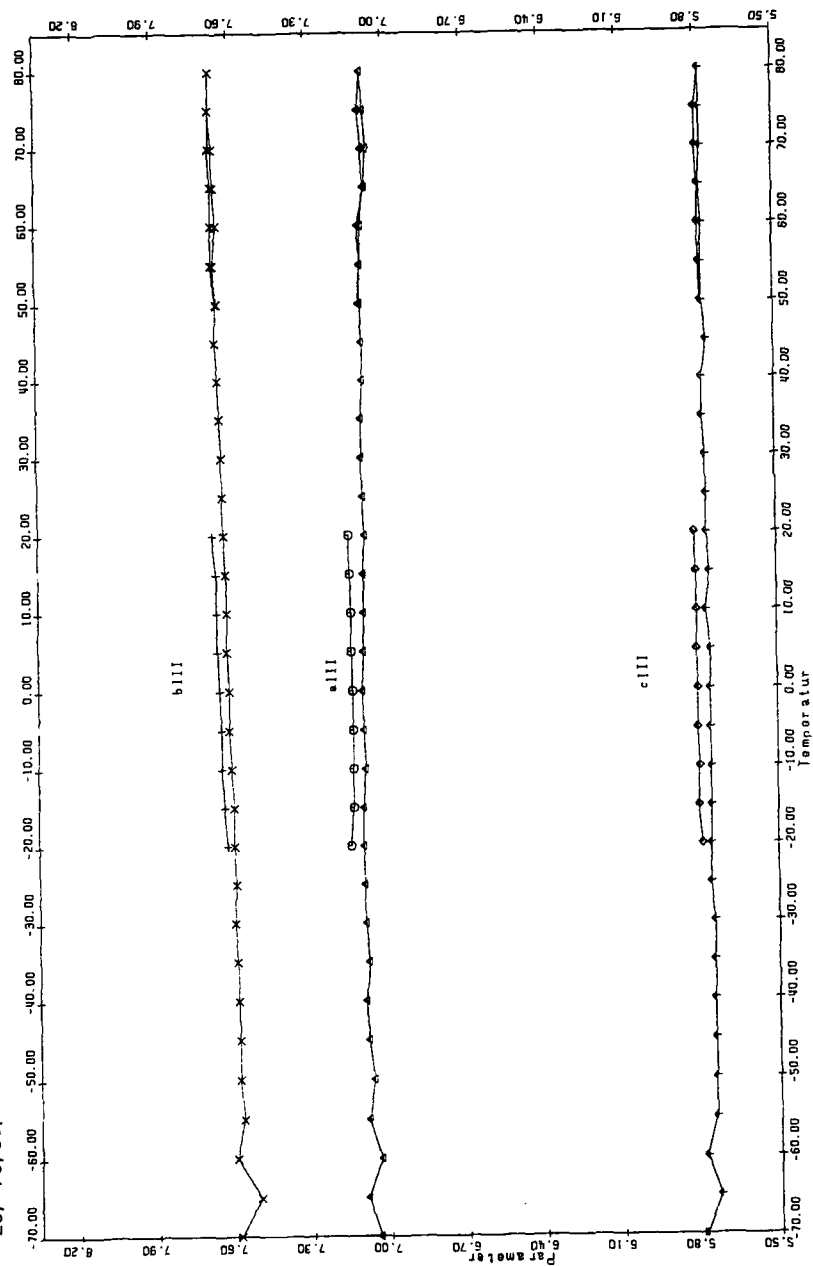
20/-70/80/-70°C



# Lattice Parameters

kf060988

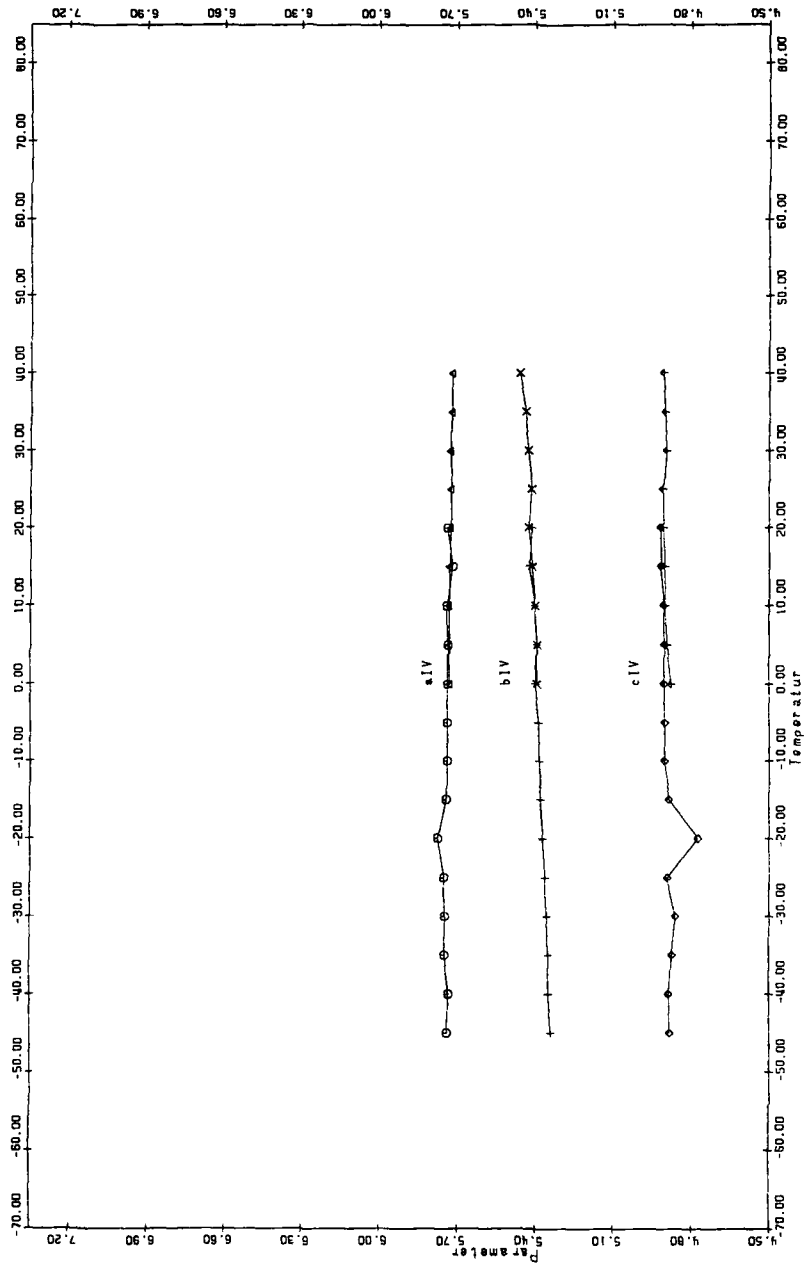
20/-70/80/-70°C



# Lattice Parameters

kf060988

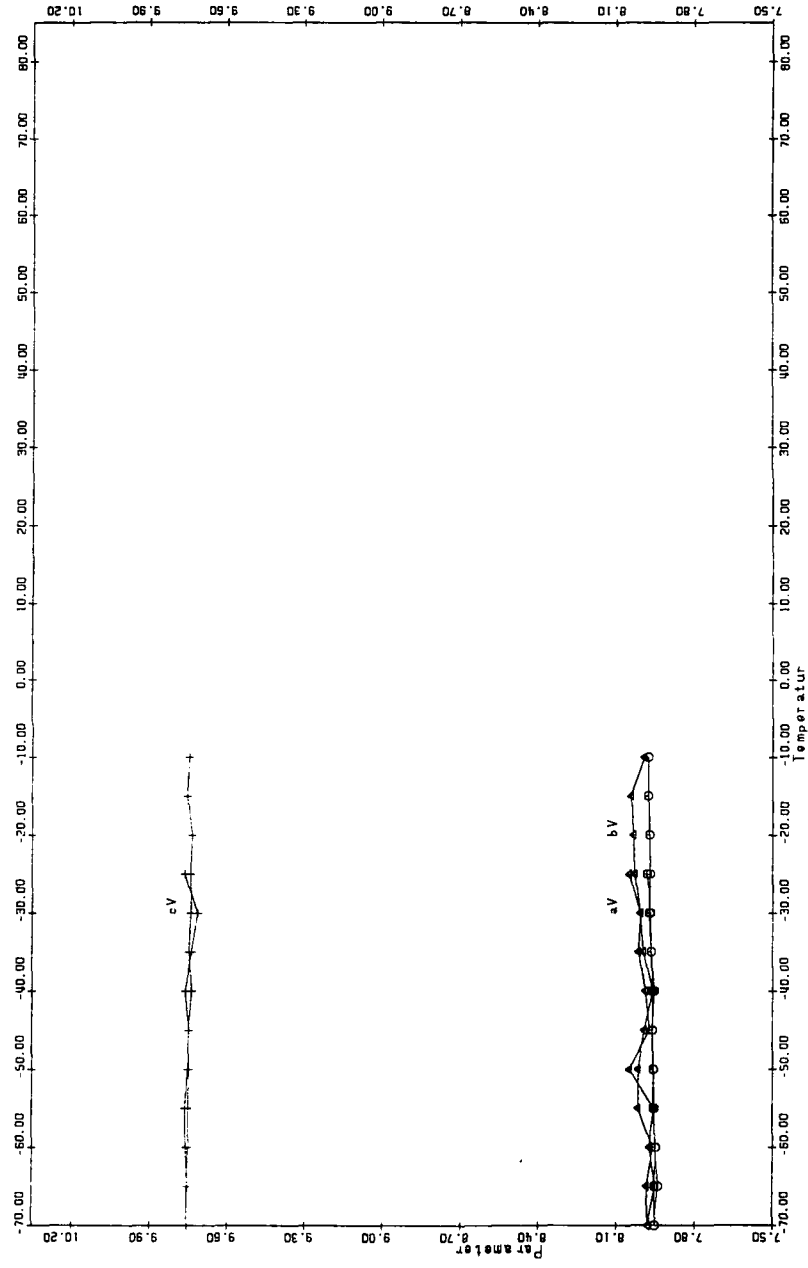
20/-70/80/-70°C



# Lattice Parameters

kf060988

20/-70/80/-70°C



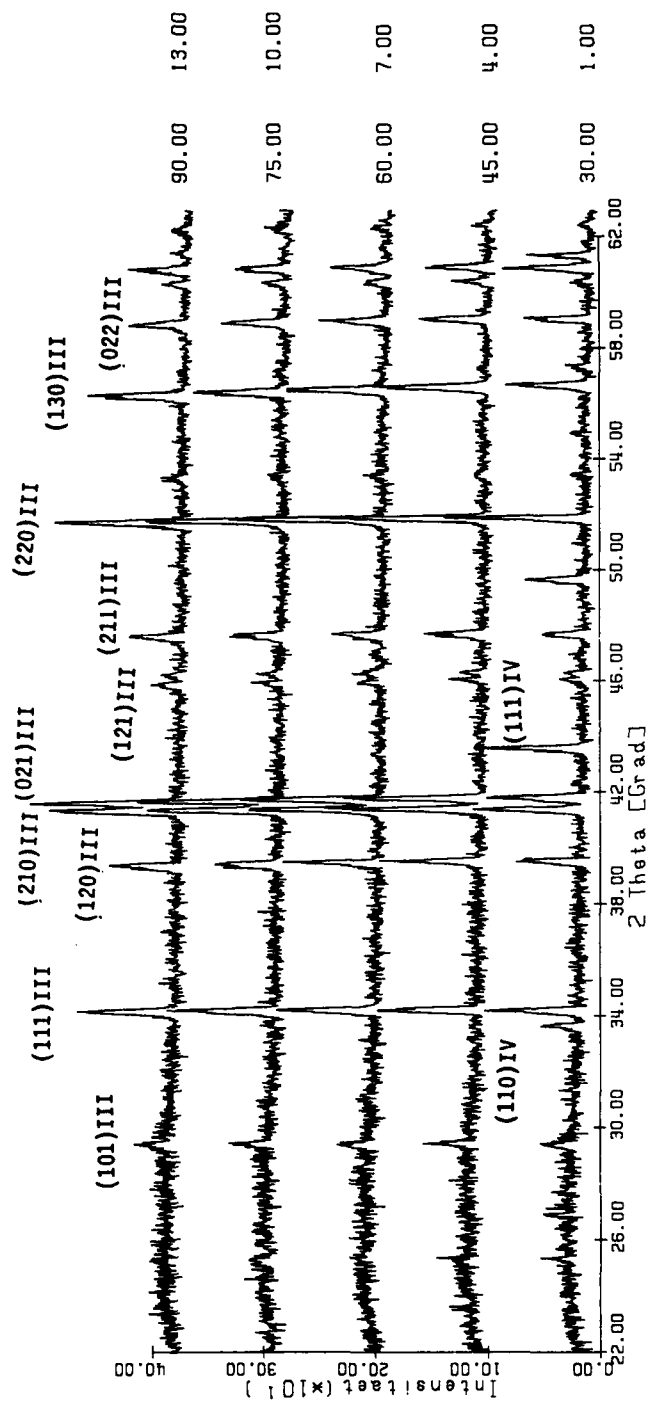
*Series*  
*KF 140988*

*Temperature*  
*Program*  
*30/140/-70/140*

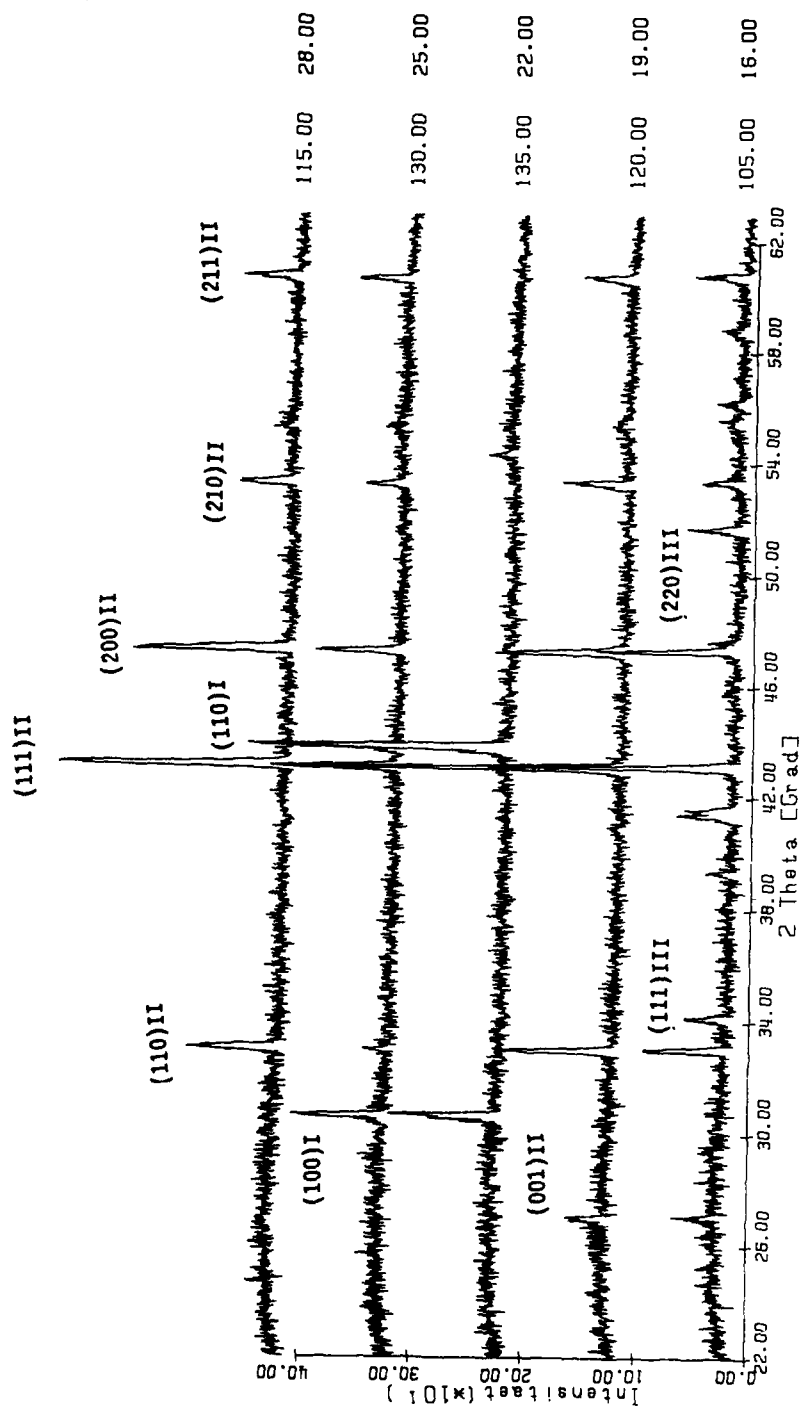
# Diffraction Patterns

k f 140988

30/140/-70/140°C



Kf140988





Intensity (x10<sup>4</sup>)

2-theta [Grad]

(110)II

(110)IV

(111)III

(111)IV

(211)III

(020)IV

(220)III

(210)II

40.00

30.00

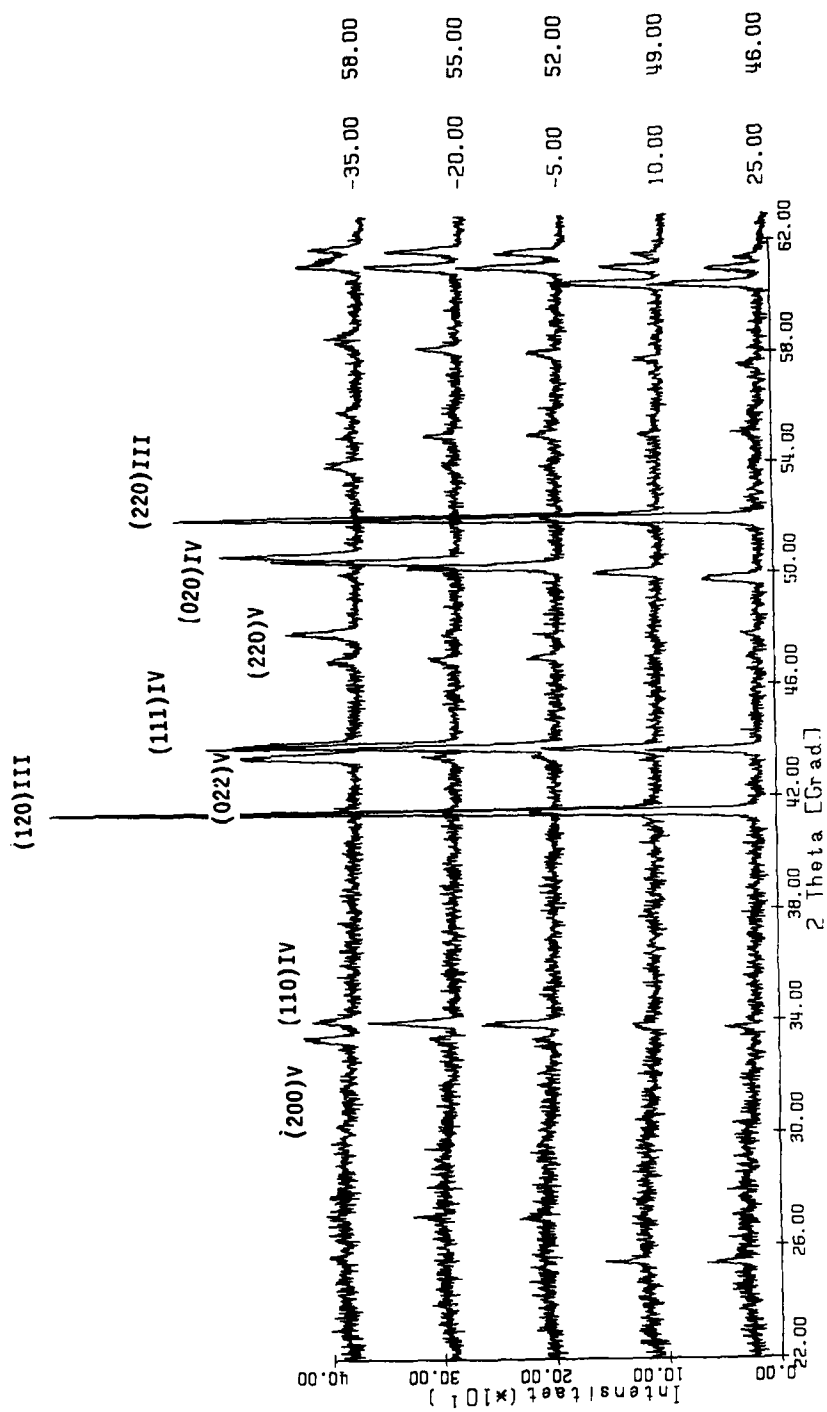
20.00

10.00

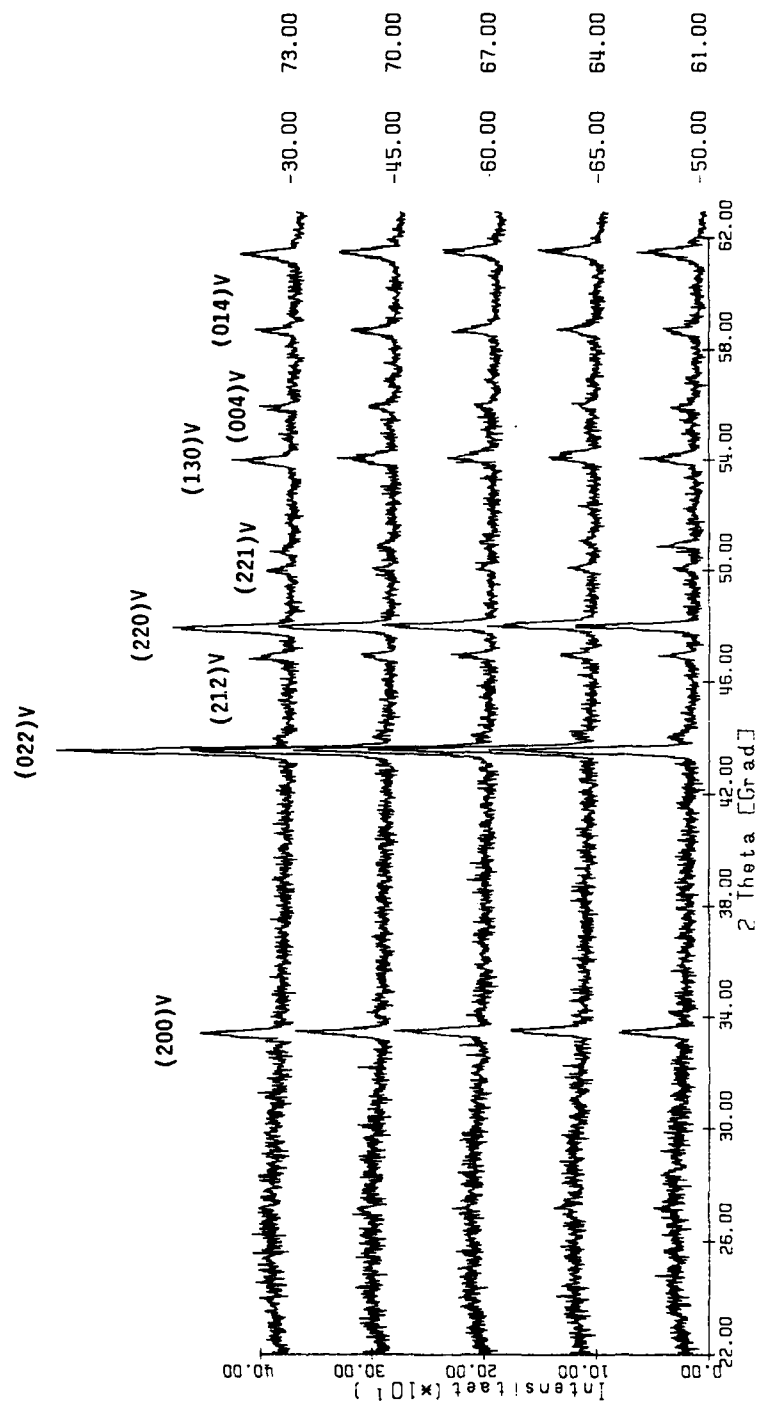
0.00

22.00 25.00 30.00 34.00 38.00 42.00 46.00 50.00 54.00 58.00 62.00

kf140988

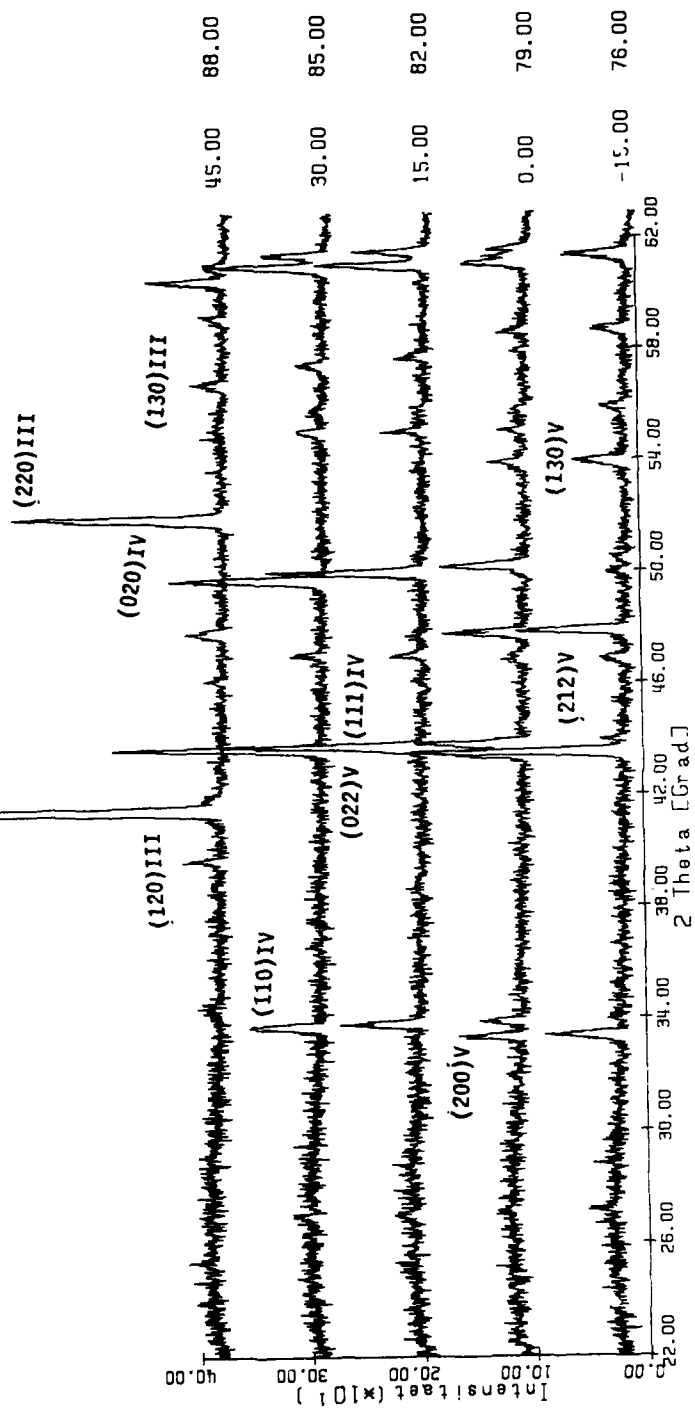


kf140988



(210) III

kfi40988

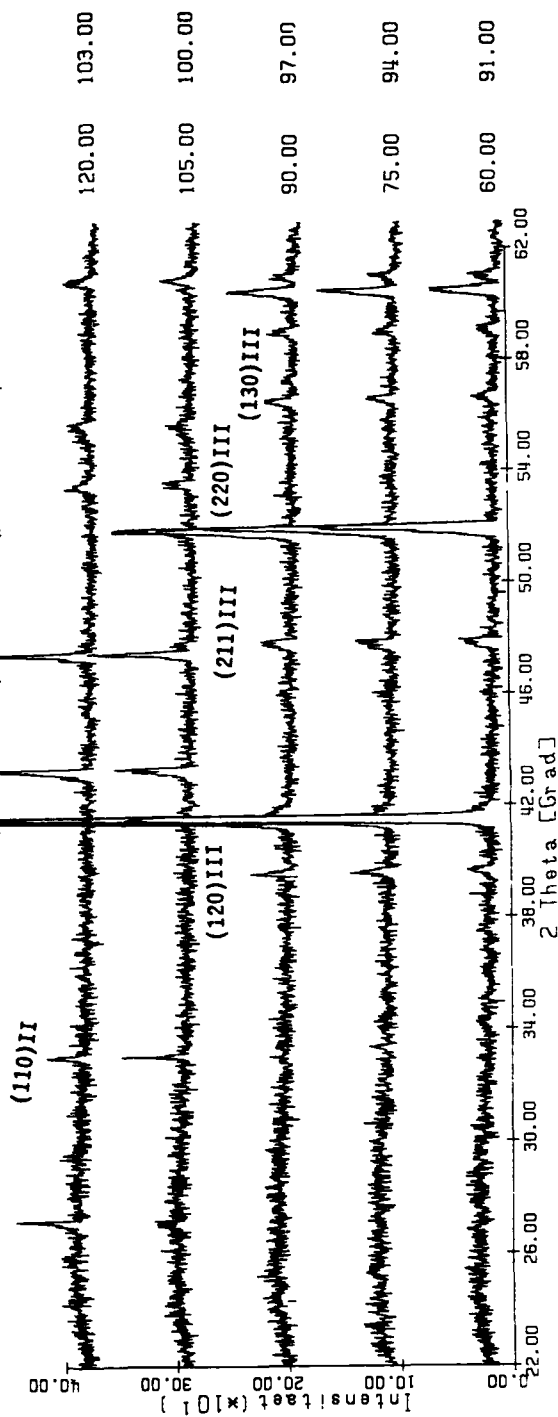


(021) III

kf140988

(001) II (100) I

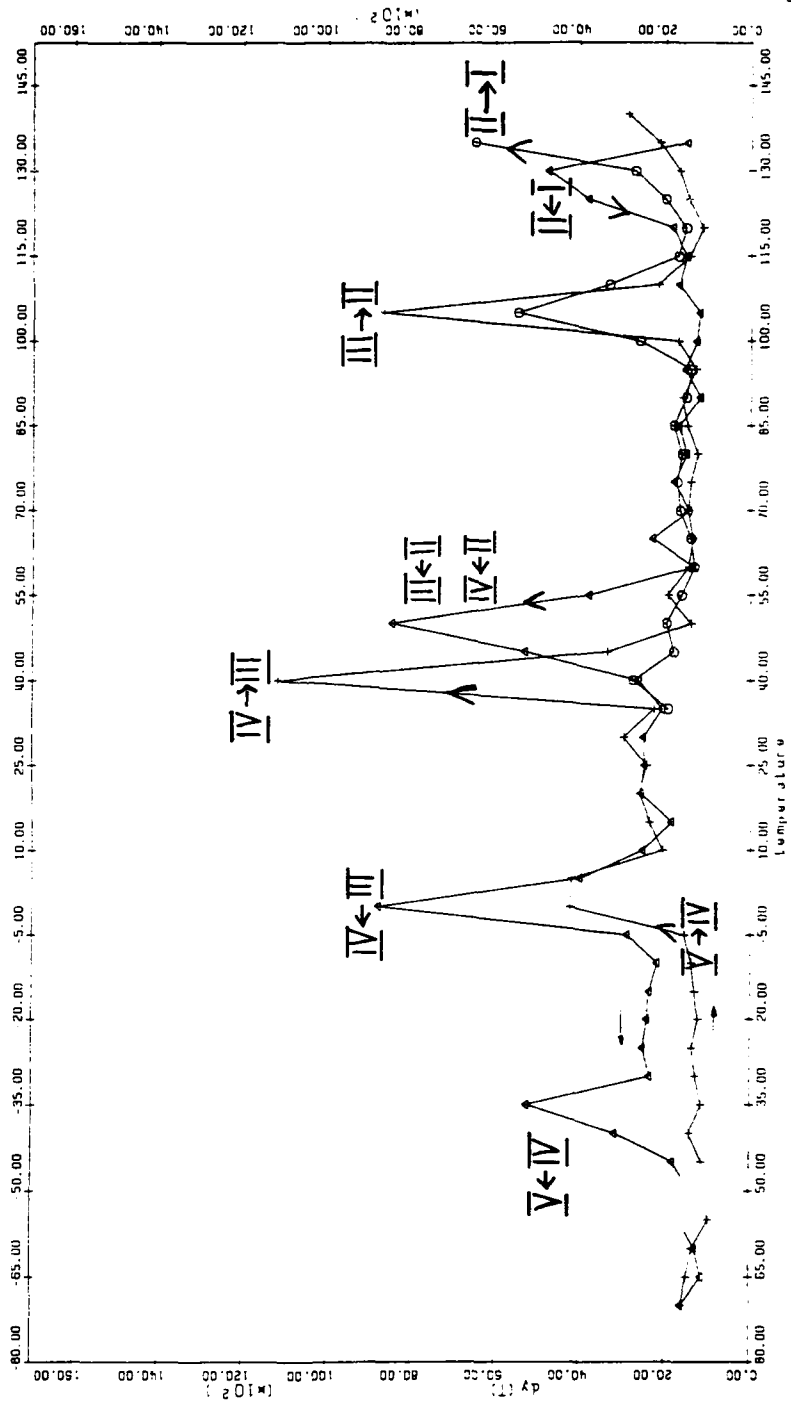
(111) II  
(110) I (200) II



# Difference Curve DY(T)

kf140988

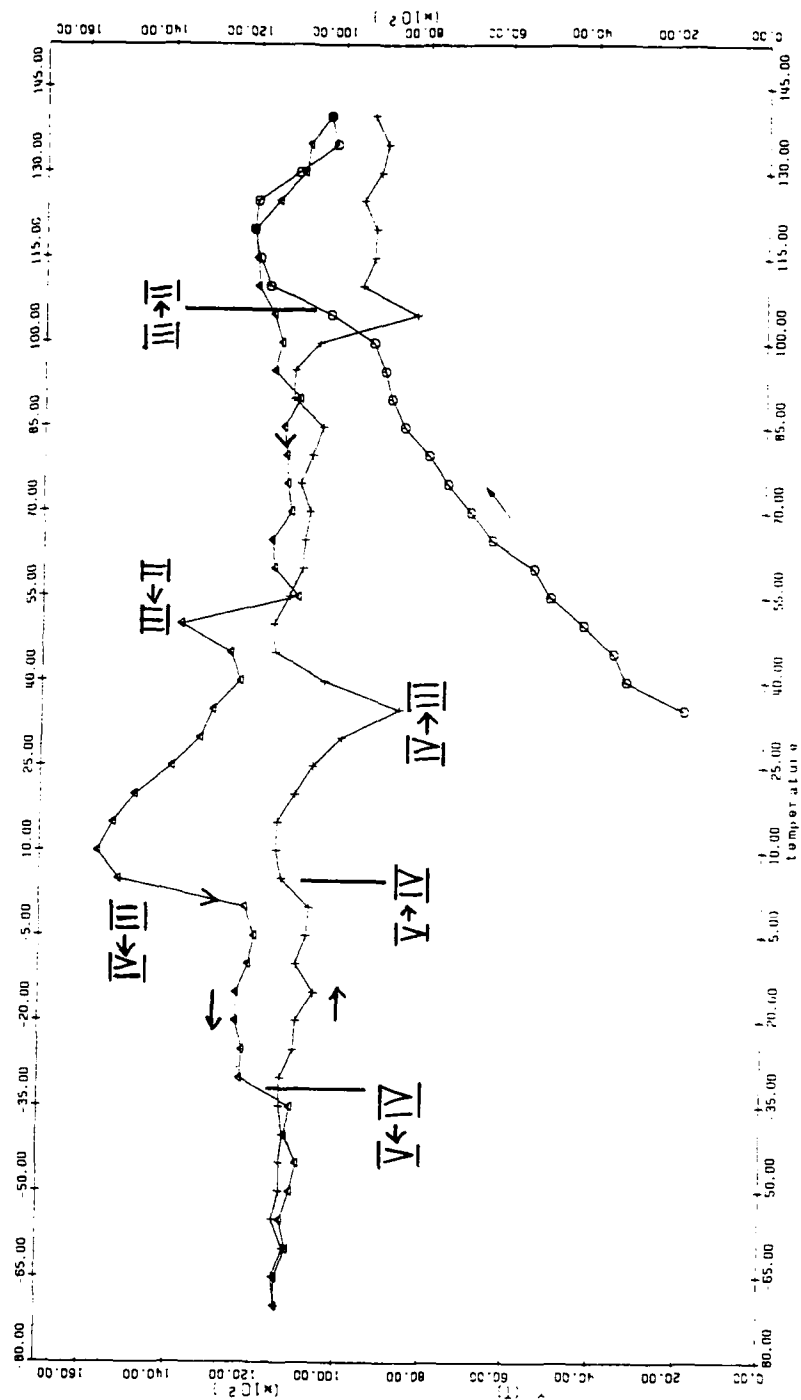
30/140/-70/140°C



# Difference Curve Y(T)

kf140988

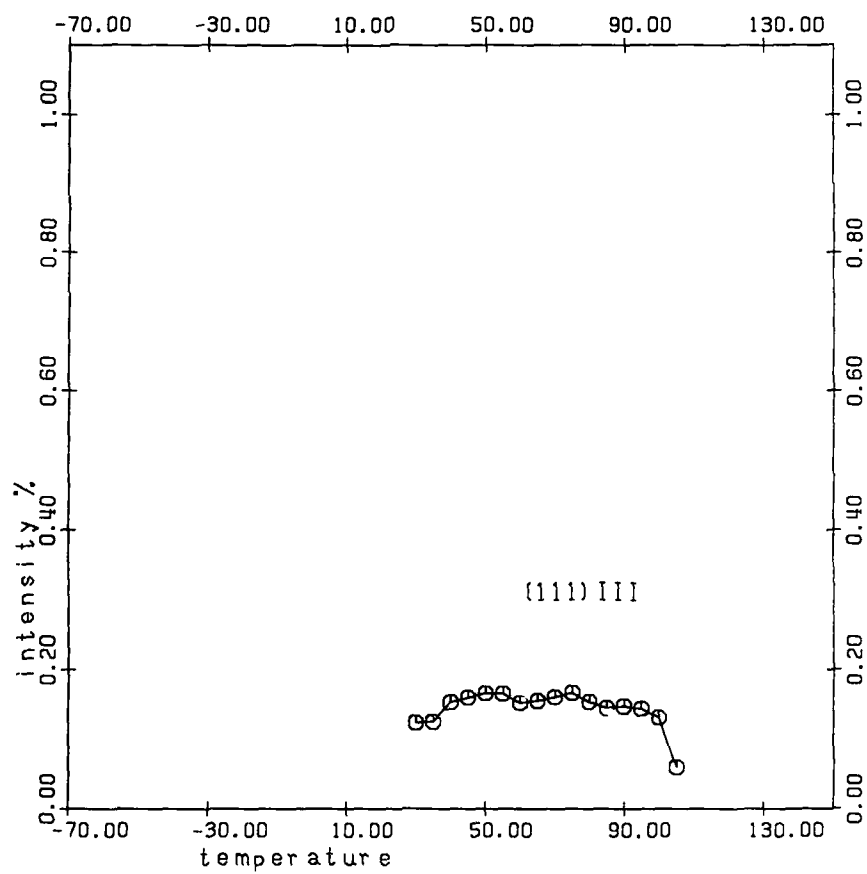
30/140/-70/140°C



# Intensities

kf140988

30/140/-70/140°C

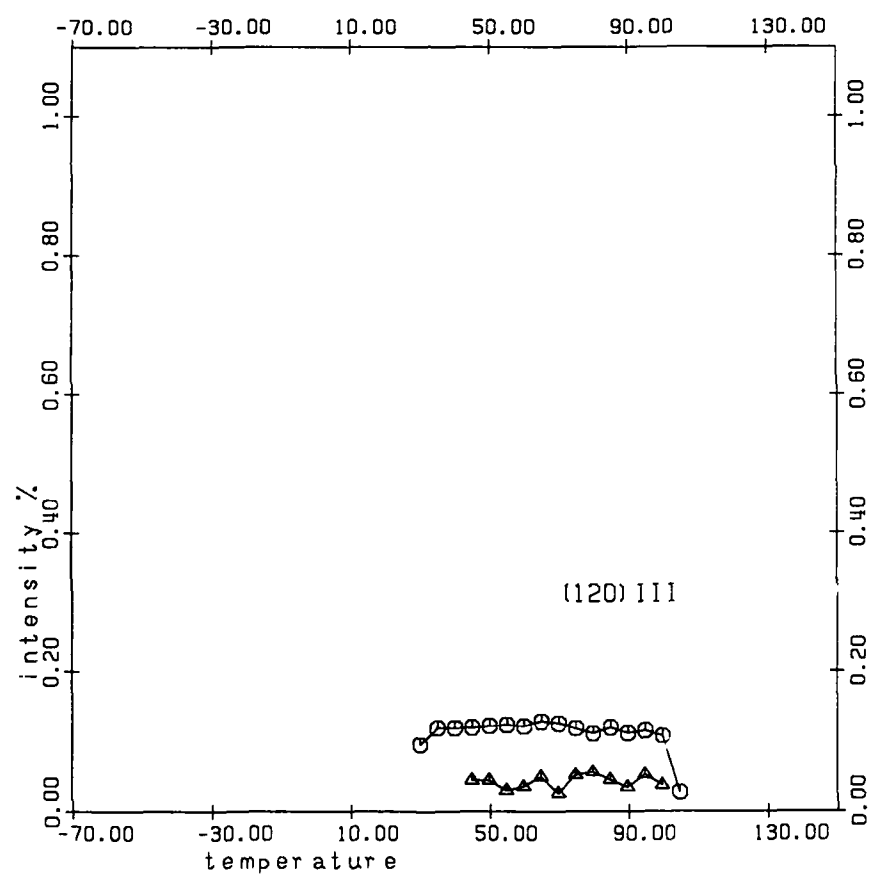




# Intensities

kf140988

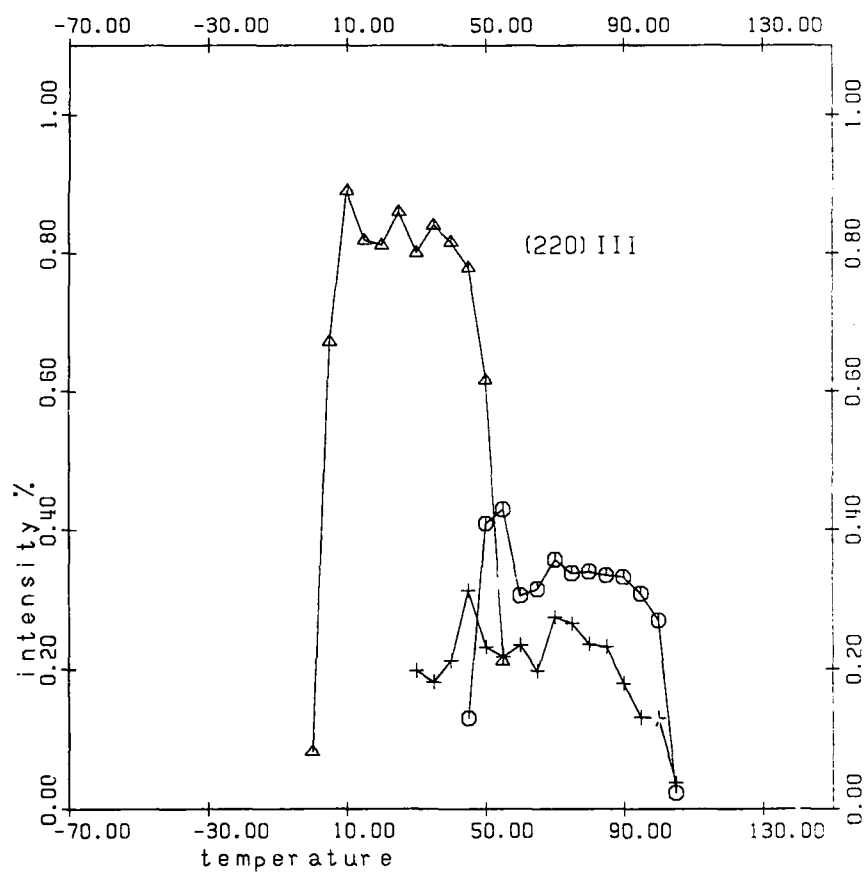
30/140/-70/140°C



# Intensities

kf140988

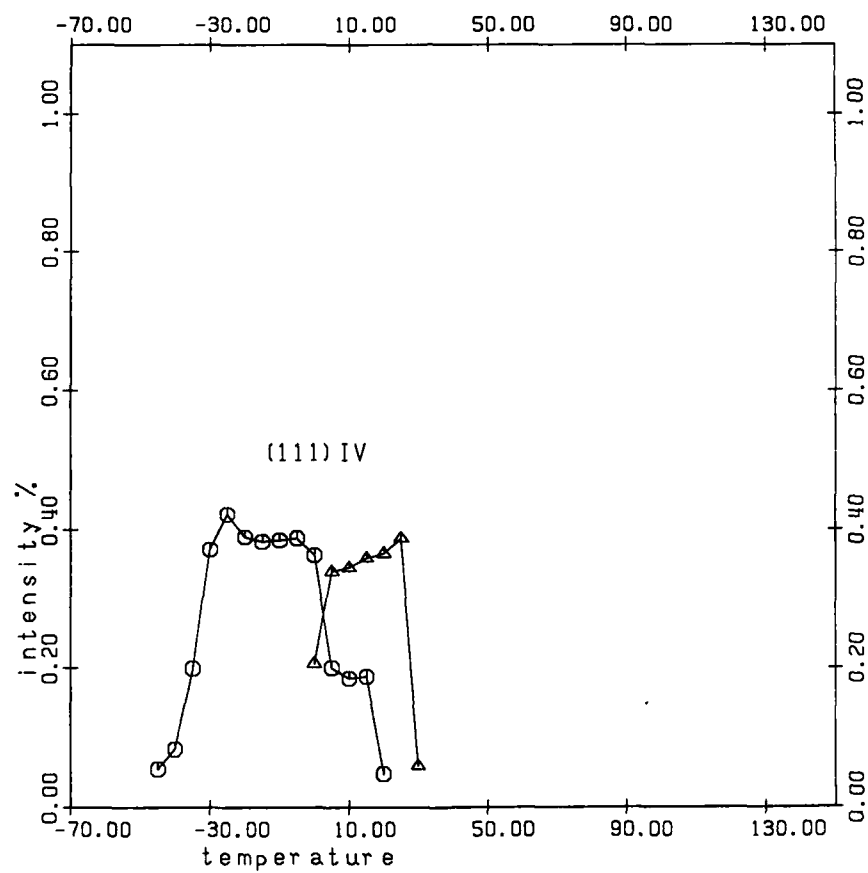
30/140/-70/140°C



# Intensities

kf140988

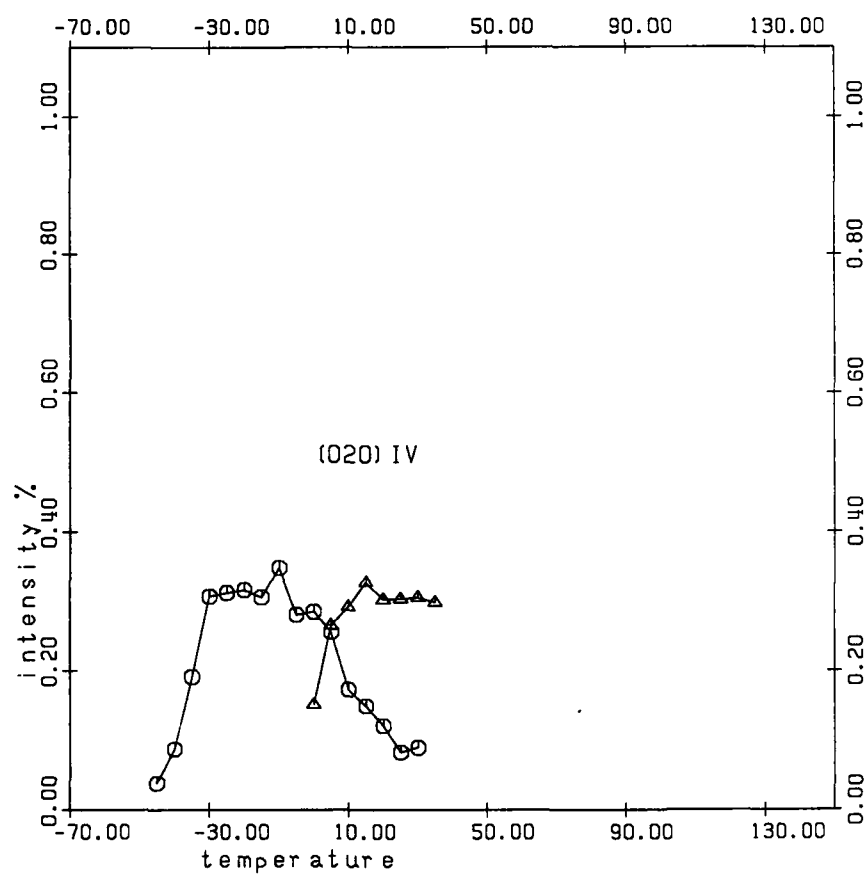
30/140/-70/140°C



# Intensities

kf140988

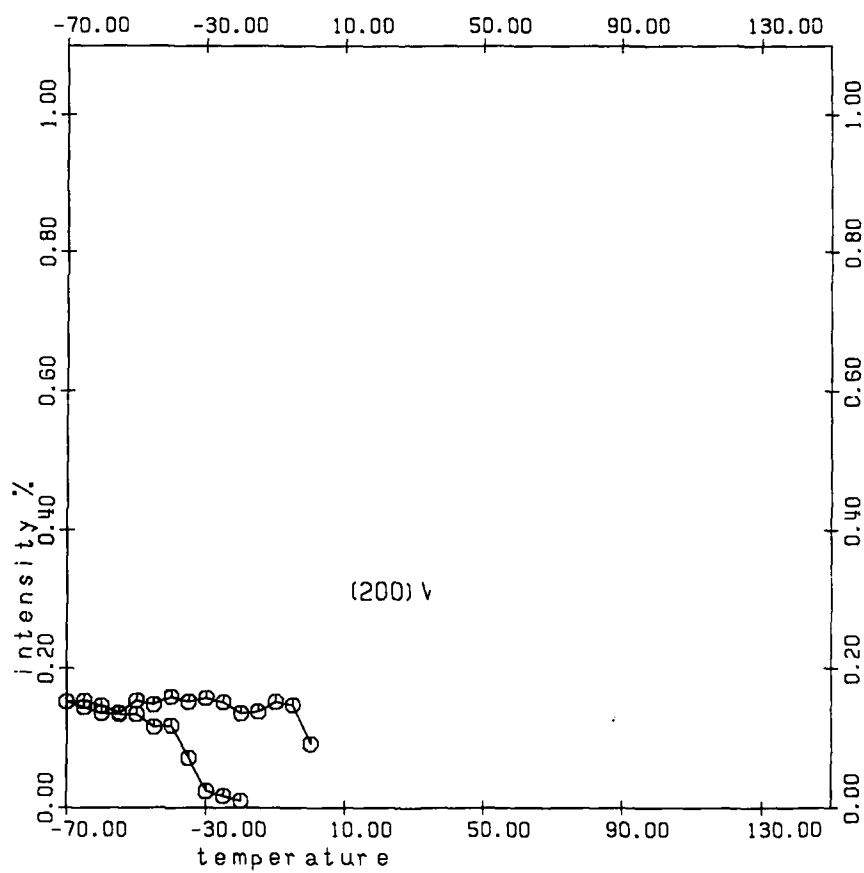
30/140/-70/140°C



# Intensities

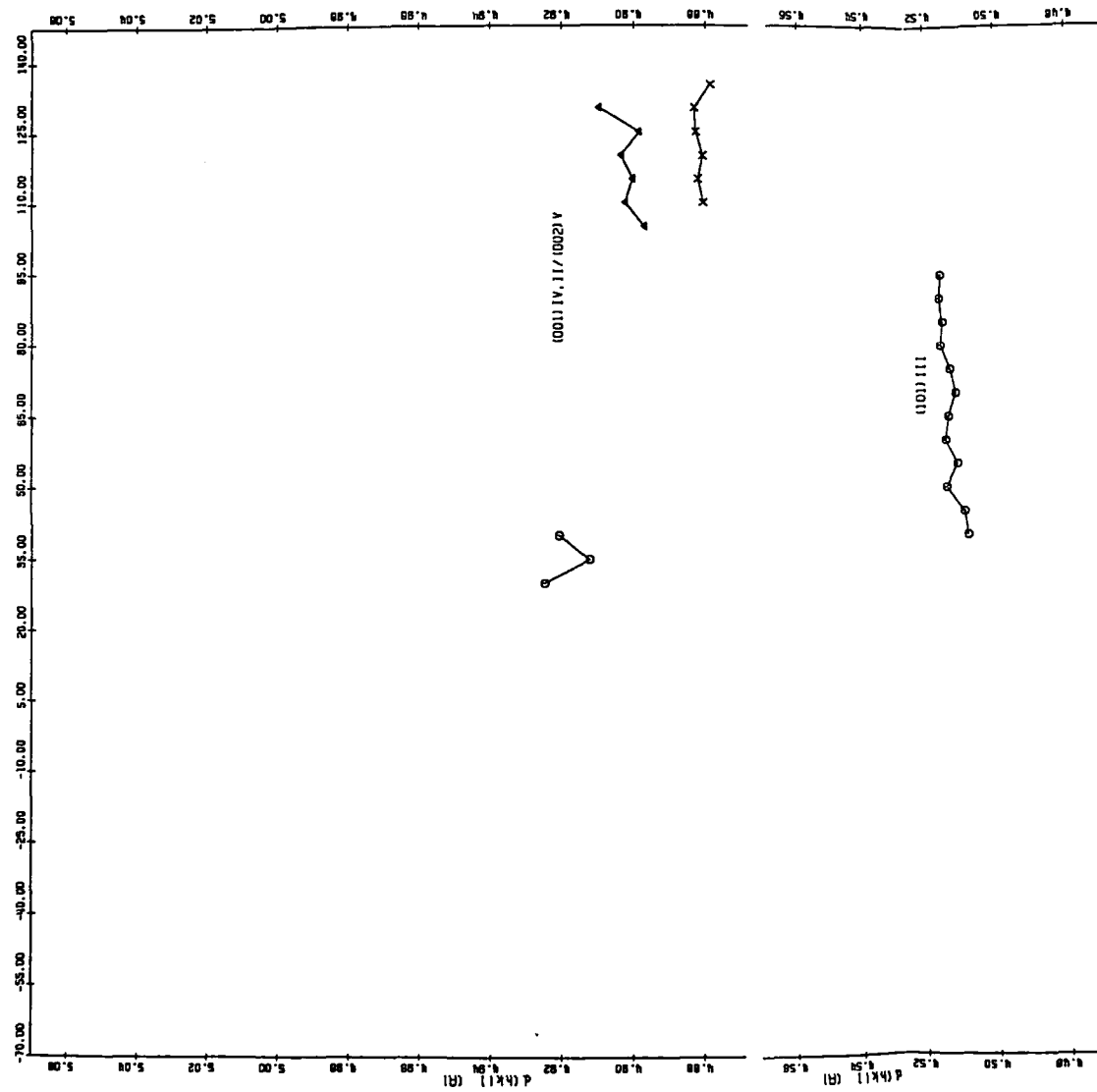
kf140988

30/140/-70/140°C



# Lattice Plane Distances

kf140988  
30/140/-70/140°C



95° 90° 85° 80° 75° 70° 65° 60° 55° 50° 45° 40° 35° 30° 25° 20° 15° 10° 5° 0°

1 (001)

11 (011) / 10 (20)

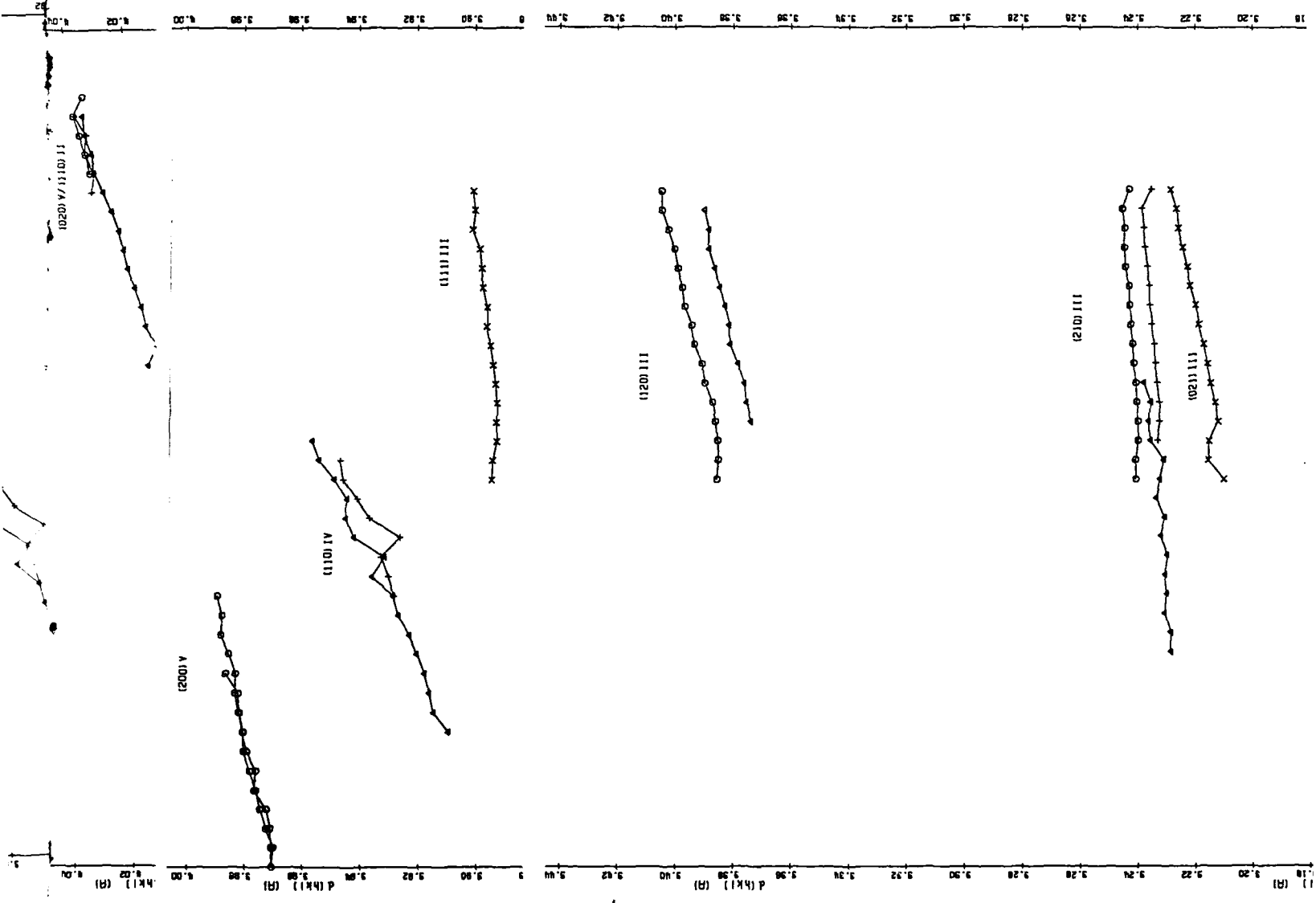
111 (101)

11 (011)

10 (02)

95° 90° 85° 80° 75° 70° 65° 60° 55° 50° 45° 40° 35° 30° 25° 20° 15° 10° 5° 0°

295

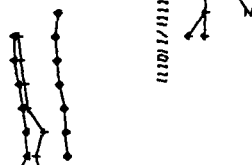


300 h

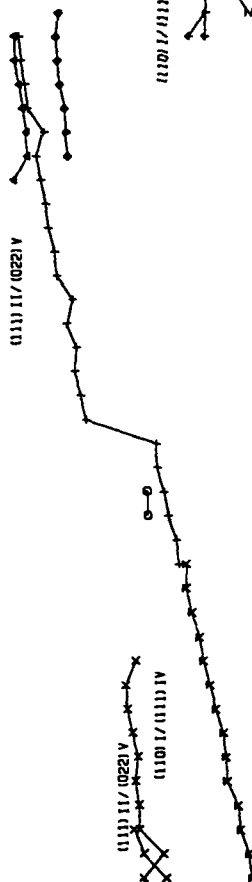


2.90 2.88 2.86 2.84 2.82 2.80 2.78 2.76 2.74 2.72 2.70 2.68 2.66 2.64 2.62 2.60 2.58 2.56 2.54 2.52 2.50

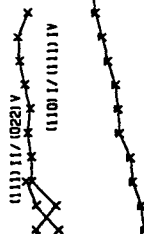
Al (111) / (1011)



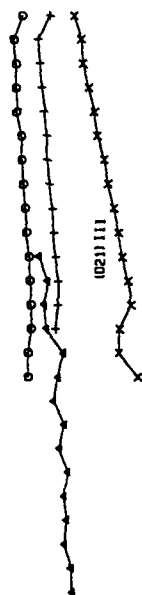
Al (111) / (0221)



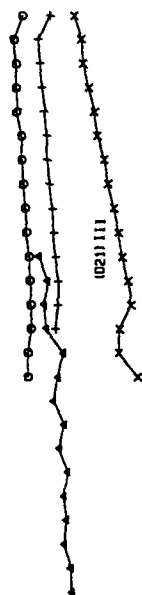
Al (111) / (1011)  
Al (220) / (111)



111 (012)



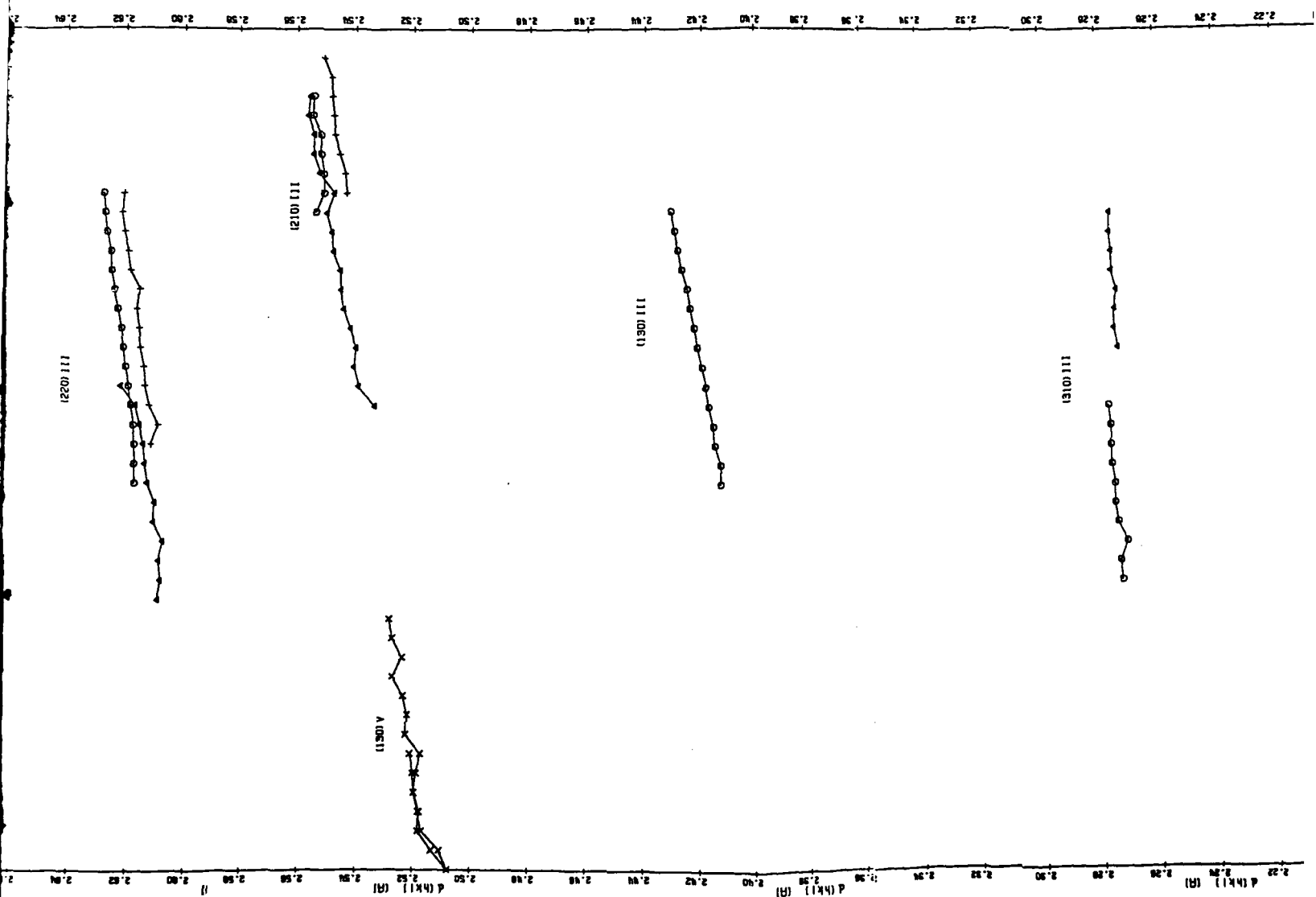
(021) (111)



2.90 2.88 2.86 2.84 2.82 2.80 2.78 2.76 2.74 2.72 2.70 2.68 2.66 2.64 2.62 2.60 2.58 2.56 2.54 2.52 2.50

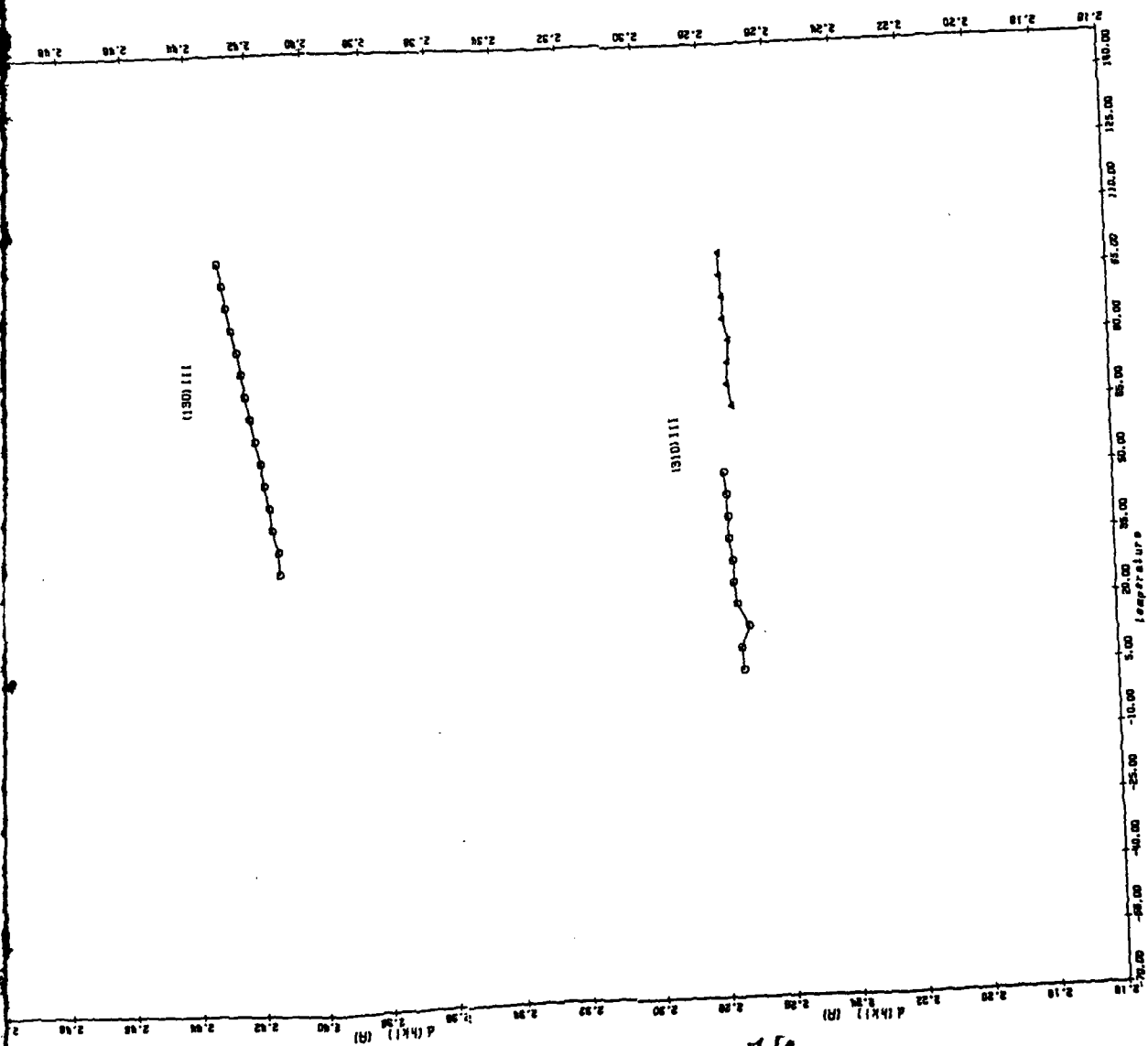
475





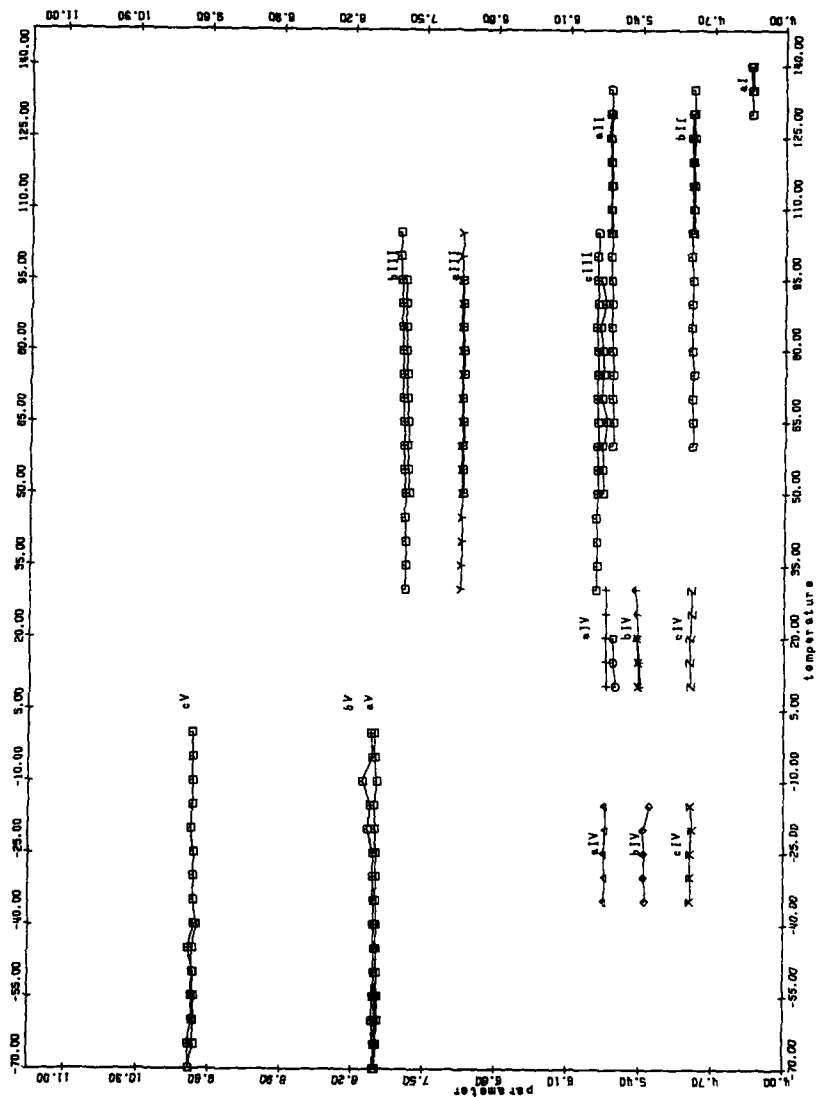
596

646



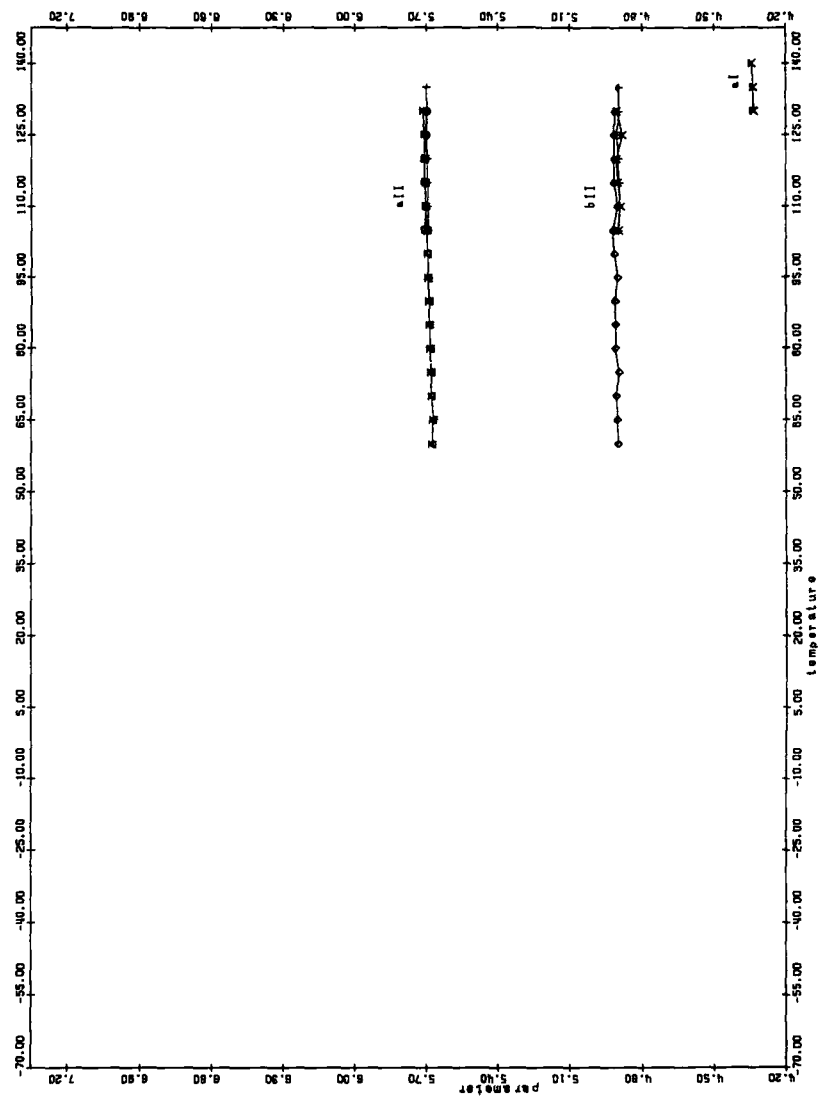
# Lattice Parameters

kf140988  
30/140/-70/140°C



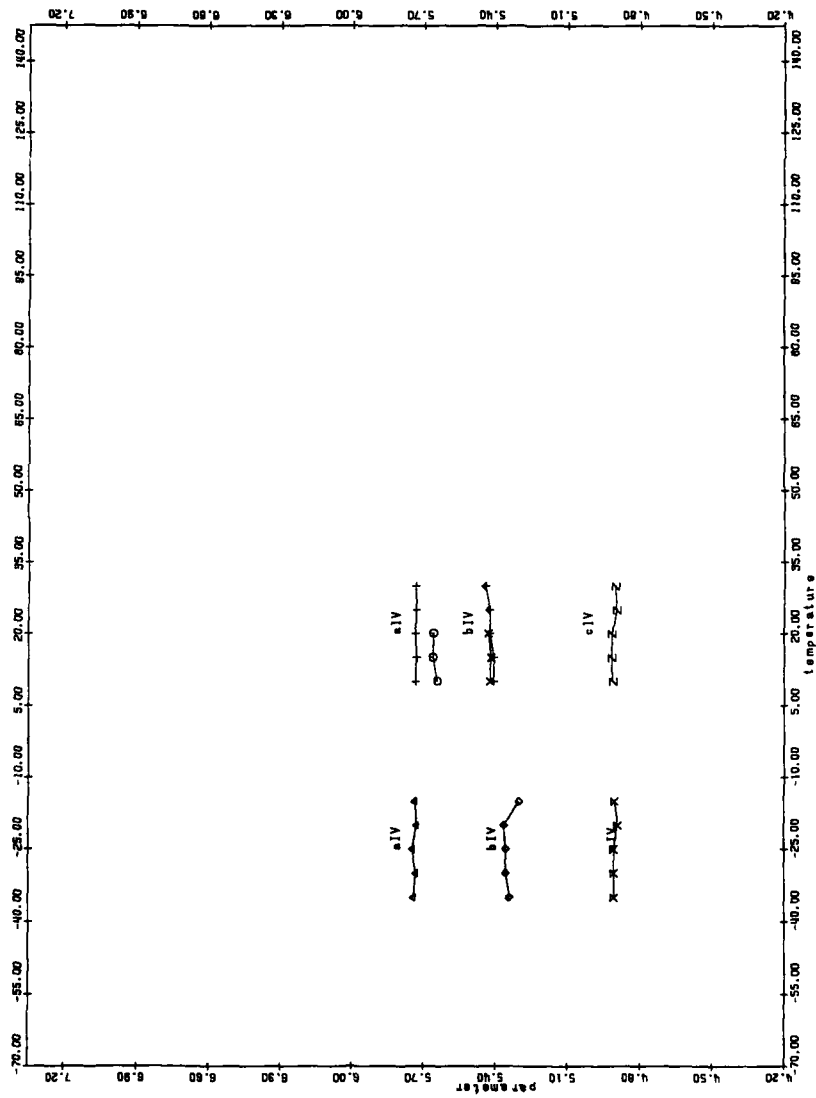
# Lattice Parameters

kf140988  
30/140/-70/140°C



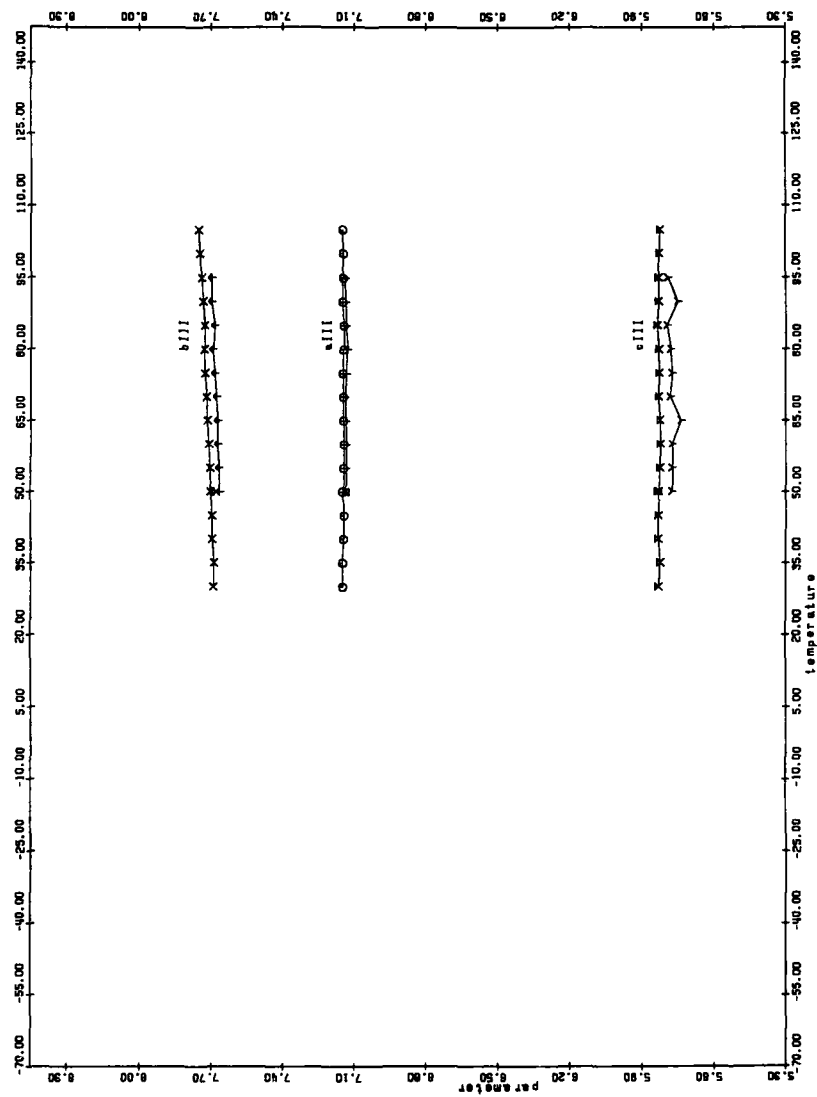
# Lattice Parameters

kf140988  
30/140/-70/140°C



# Lattice Parameters

kf140988  
30/140/-70/140°C

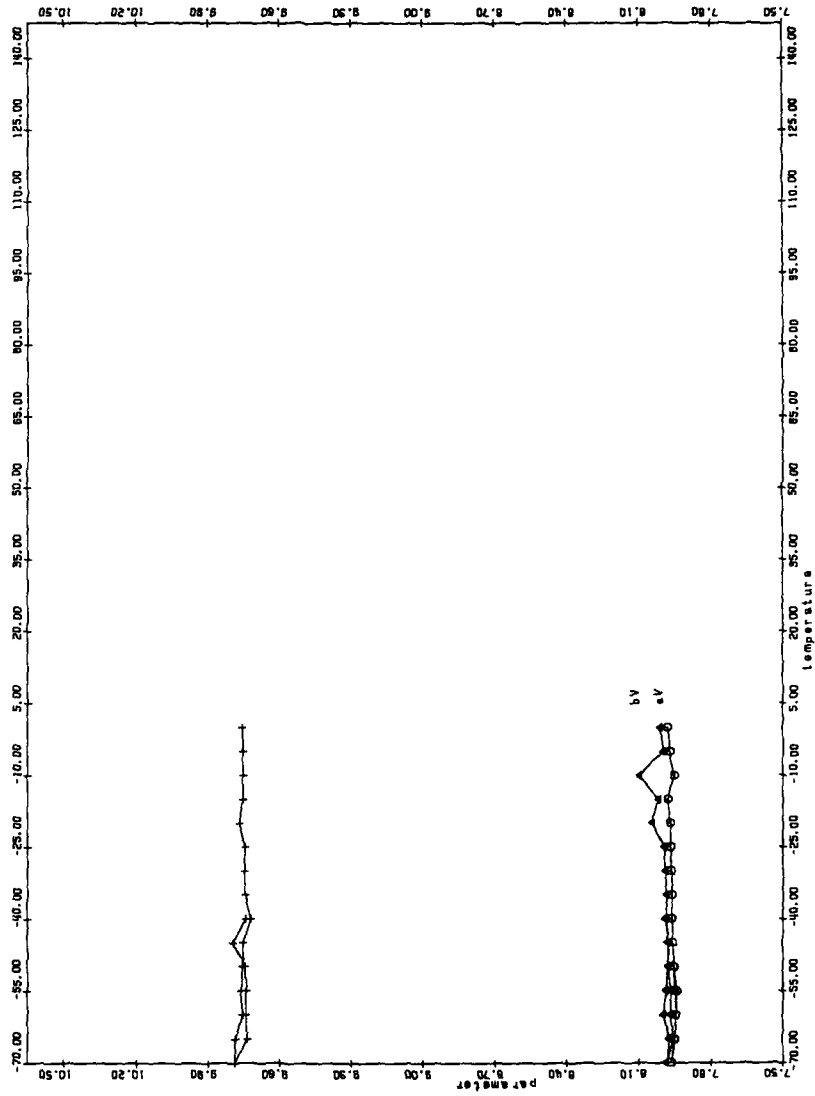




# Lattice Parameters

kf140988

30/140/-70/140°C



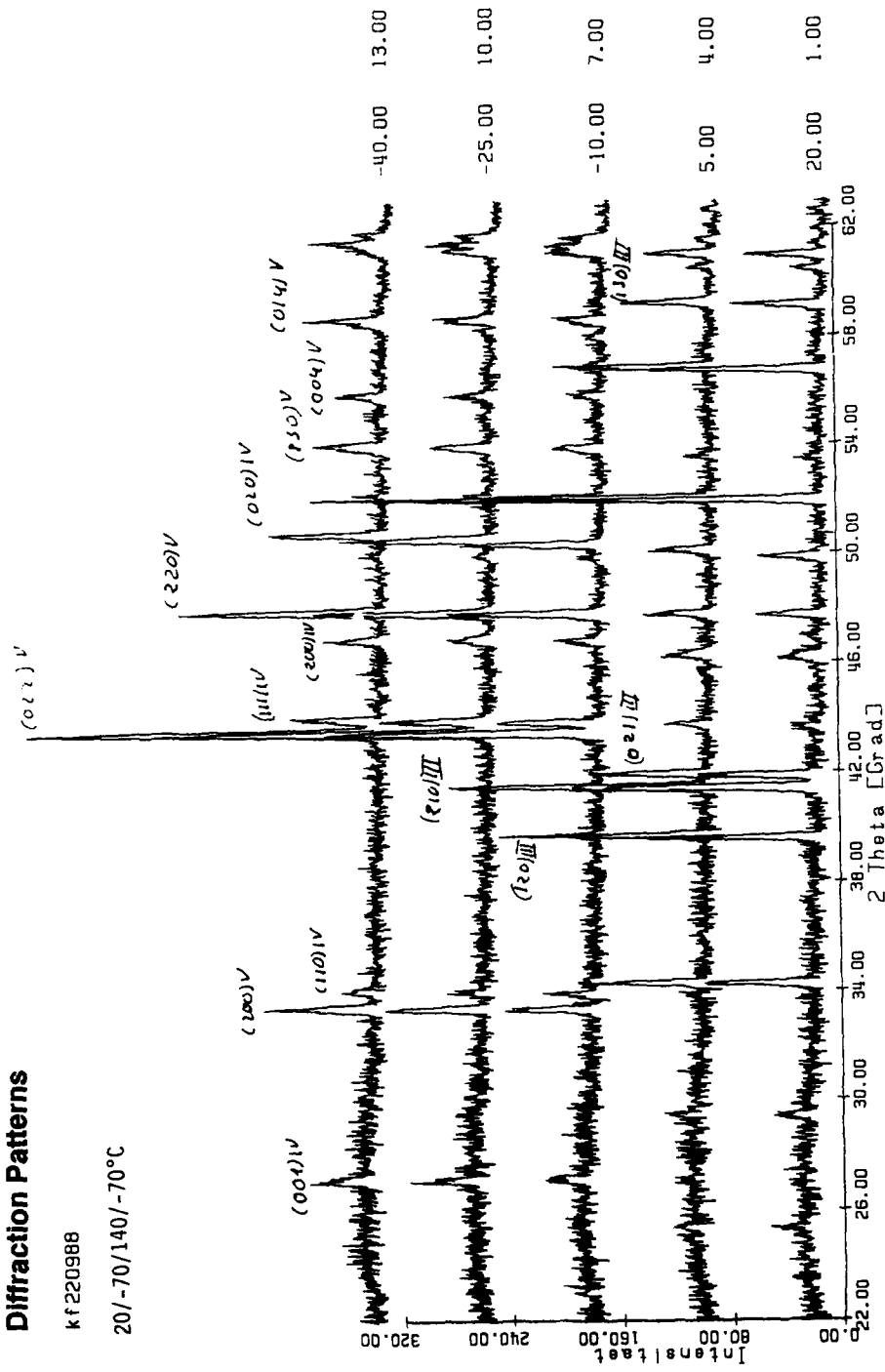
*Series*  
*KF 220988*

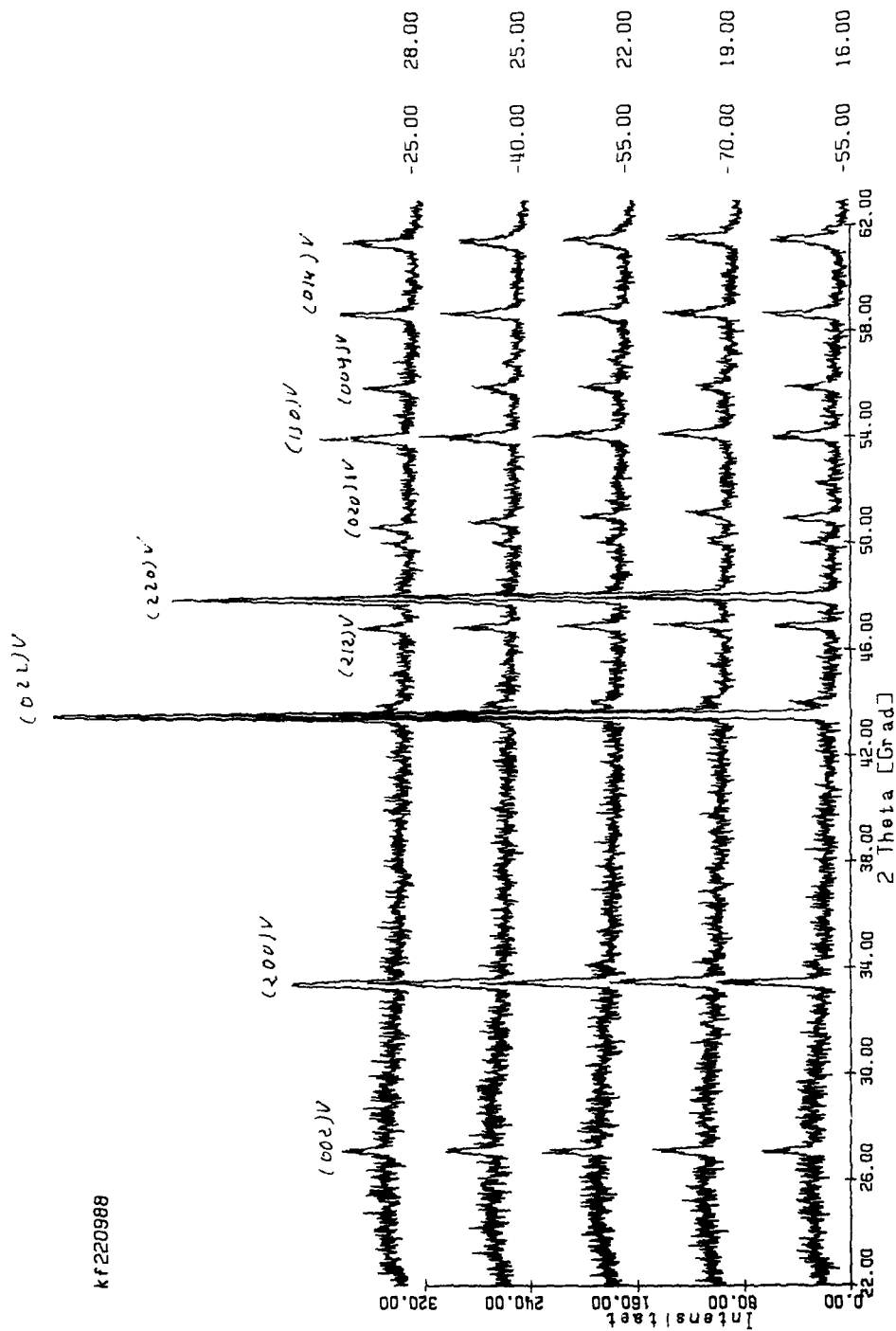
*Temperature*  
*Program*  
*20/-70/140/-70*

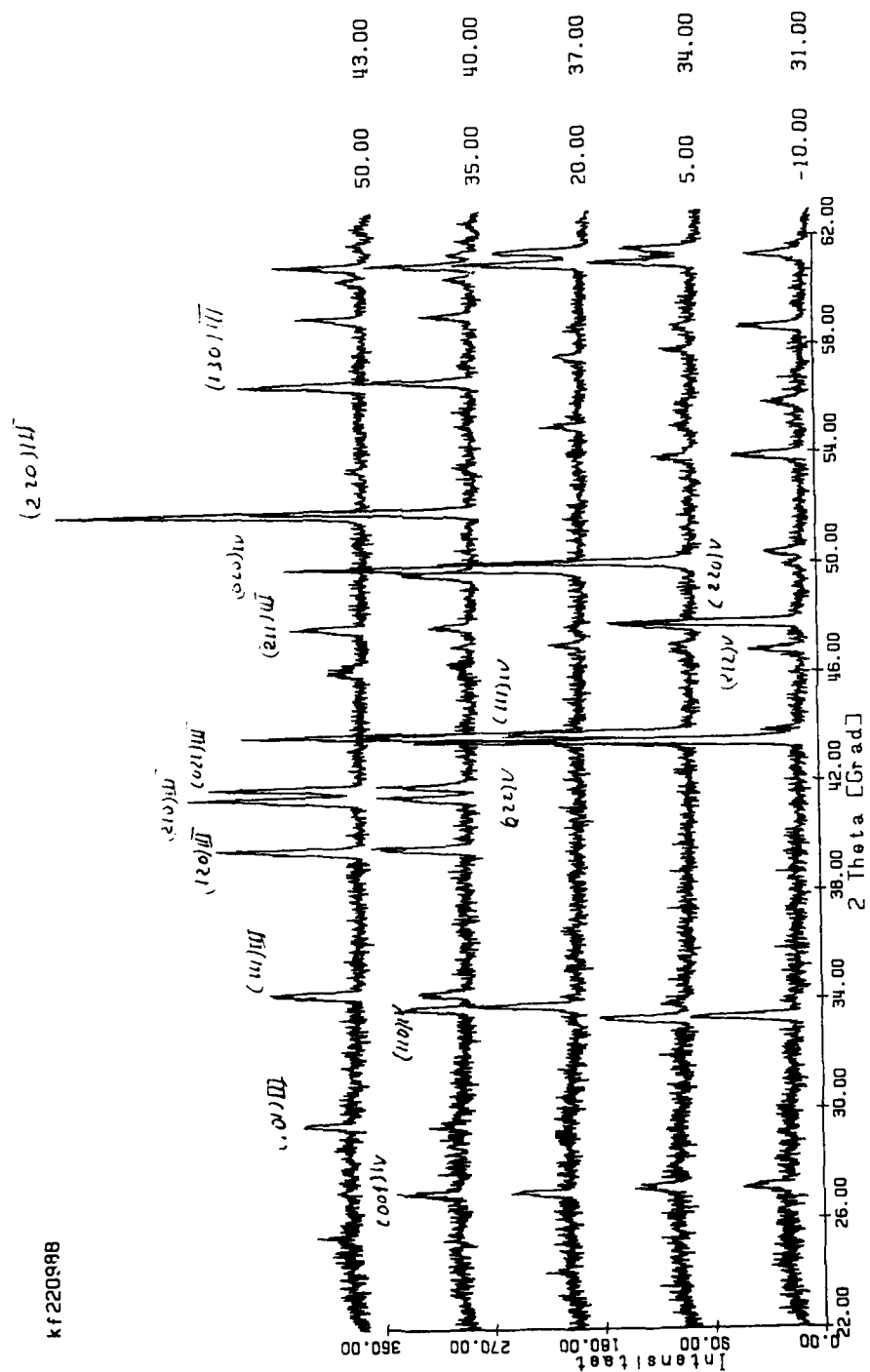
# Diffraction Patterns

Kf220988

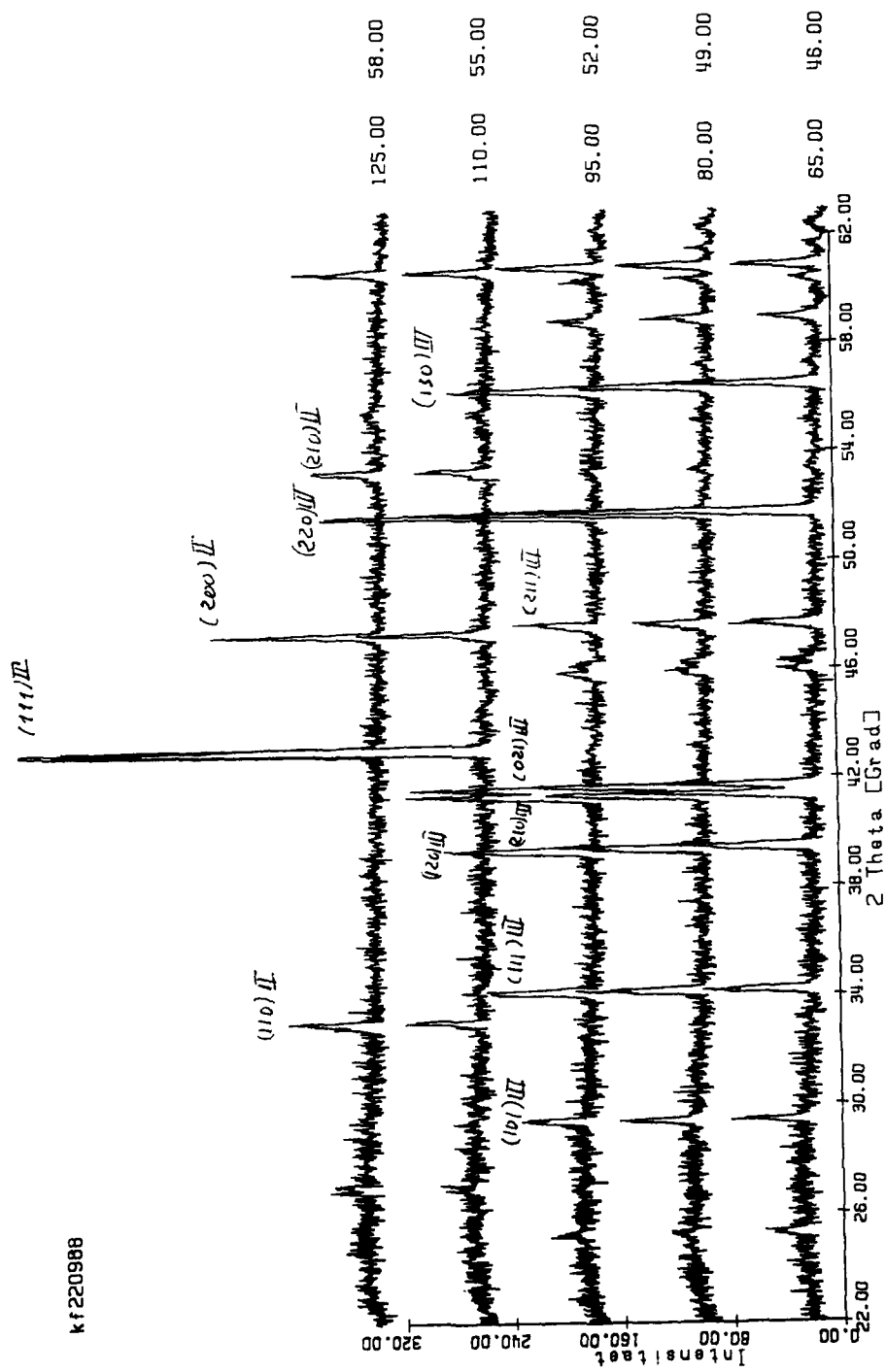
20/-70/140/-70°C

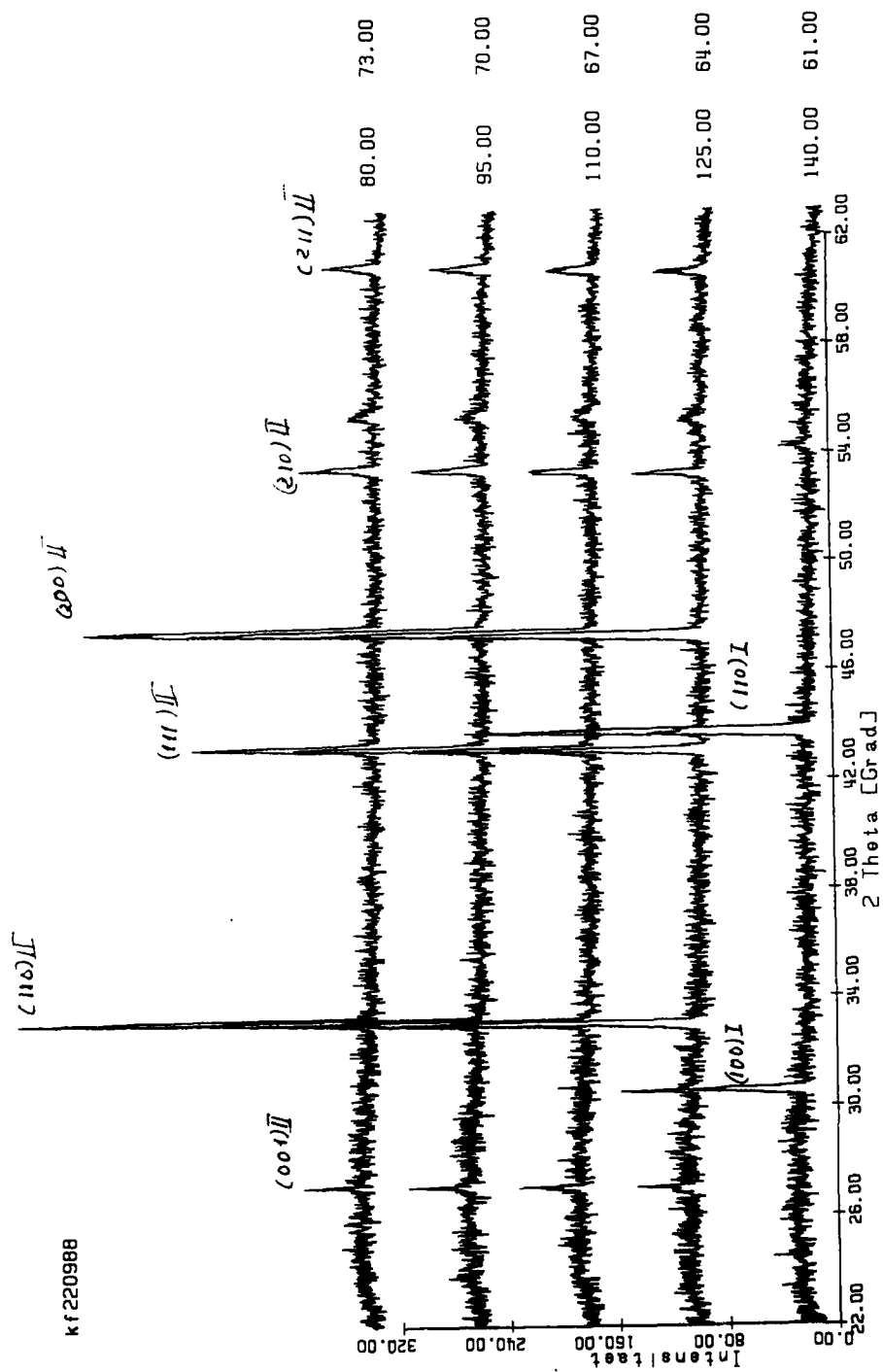


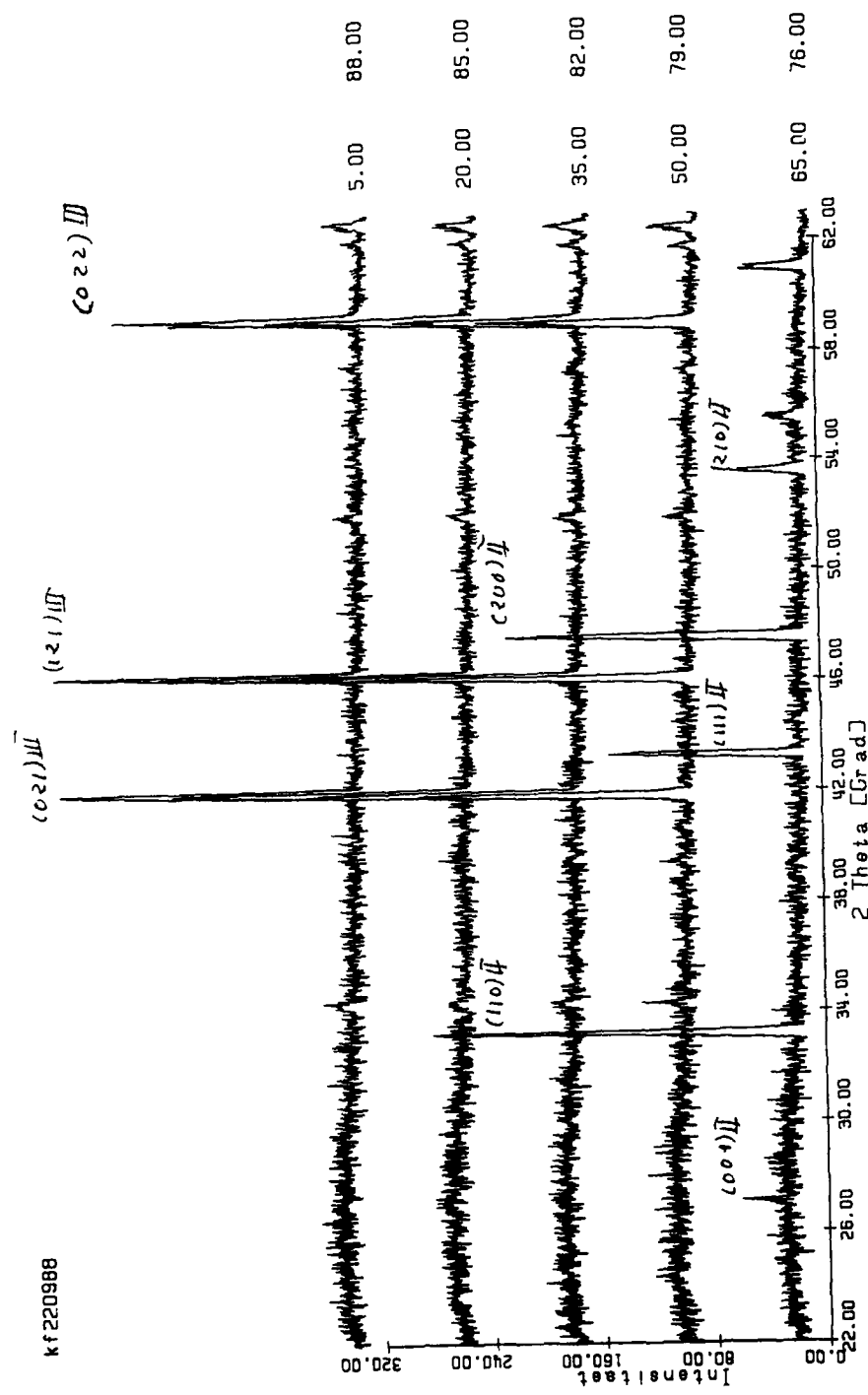




kf220988









Intensity

320.00  
240.00  
160.00  
80.00  
0.00

22.00 26.00 30.00 34.00 38.00 42.00 46.00 50.00 54.00 58.00 62.00

2 Theta [Grad]

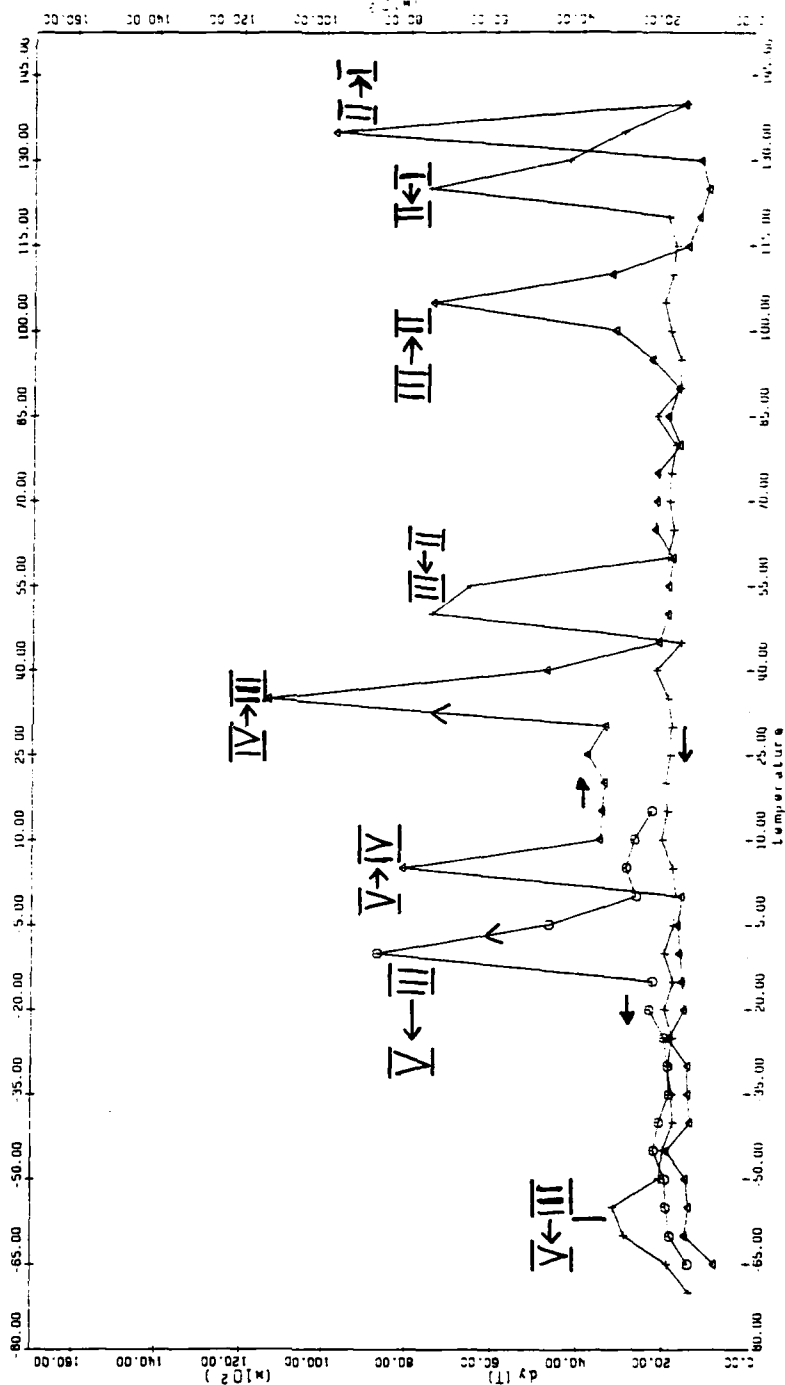
(021) III  
(021) V  
(111) III  
(111) V  
(202) III

100%  
80%  
60%  
40%  
20%

# Difference Curve DY(T)

kf220988

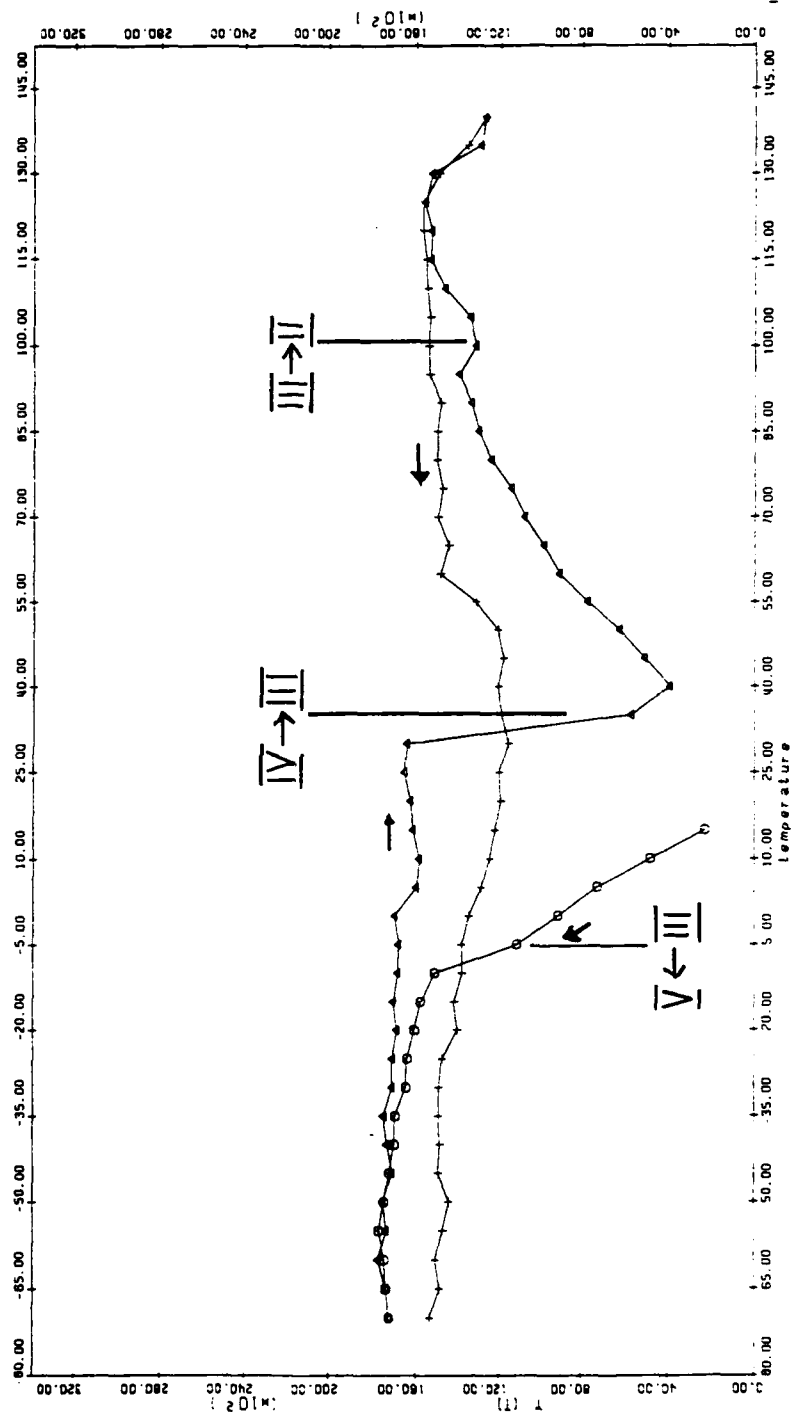
20/-70/140/-70°C



# Difference Curve Y(T)

kf220988

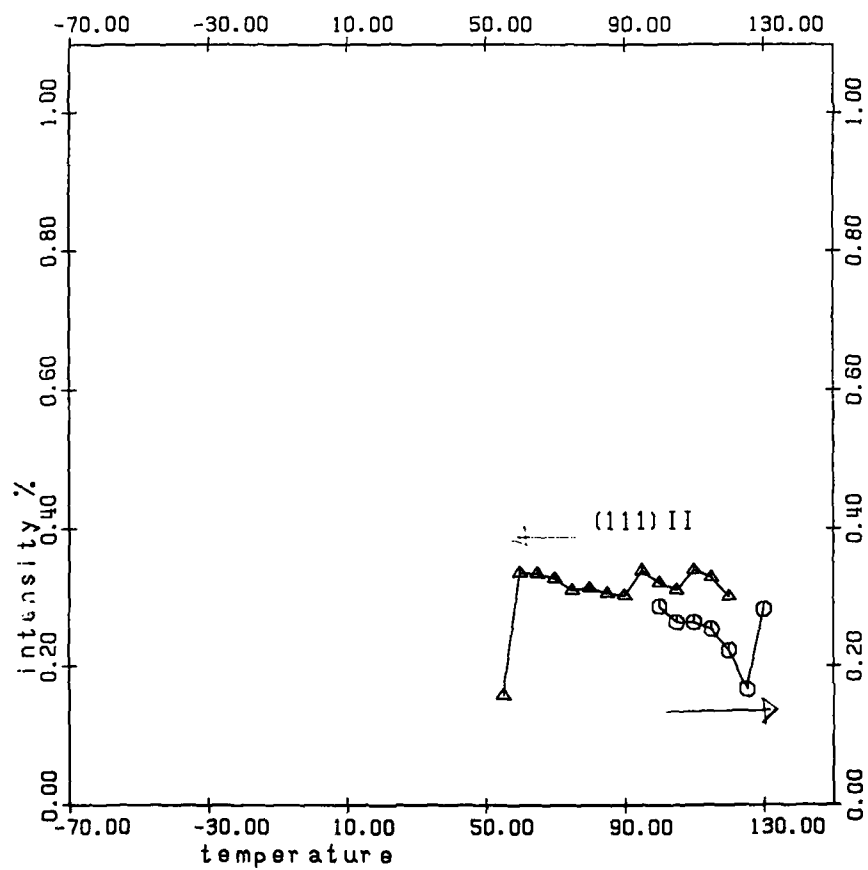
20/-70/140/-70°C



# Intensities

kf220988

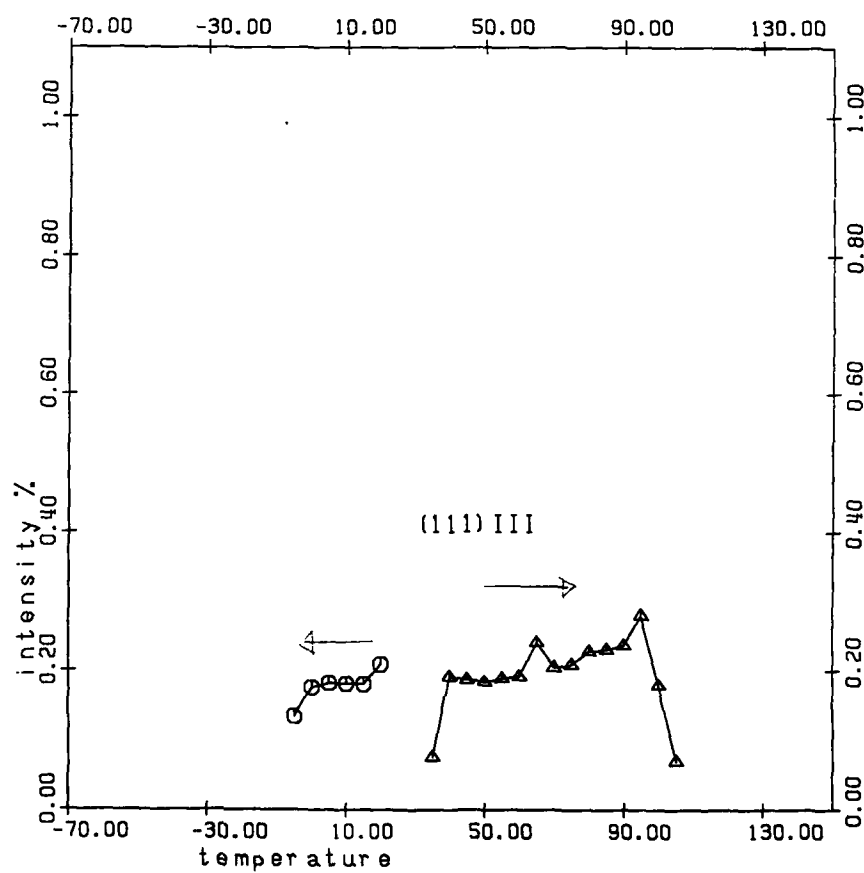
20/-70/140/-70°C



# Intensities

kf220988

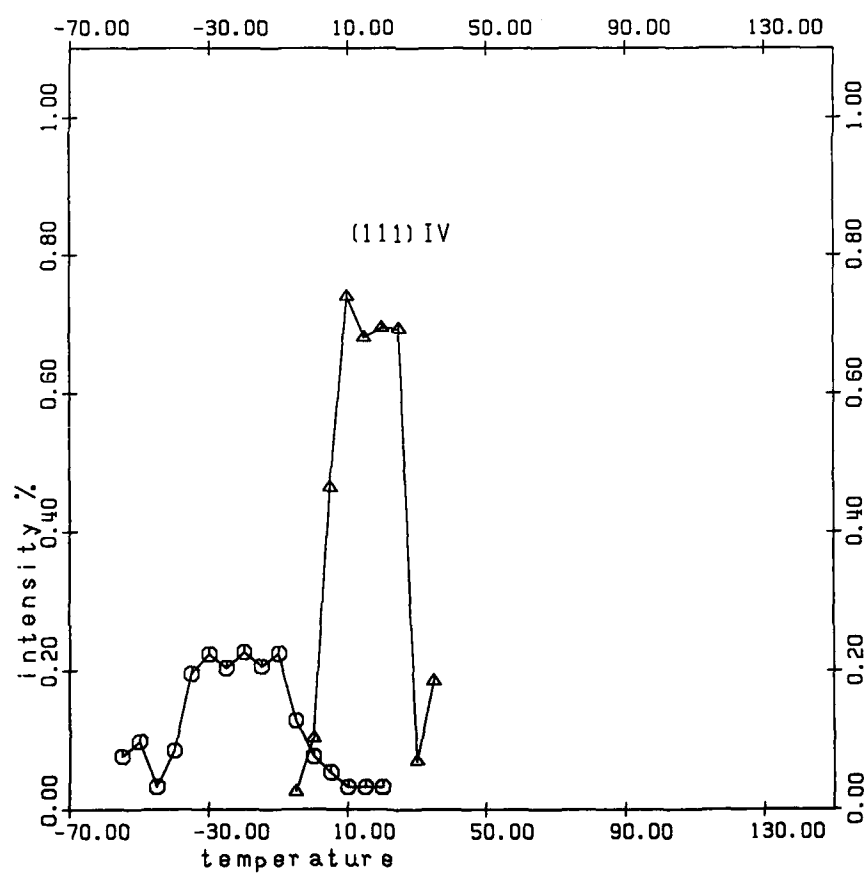
20/-70/140/-70°C



# Intensities

kf220988

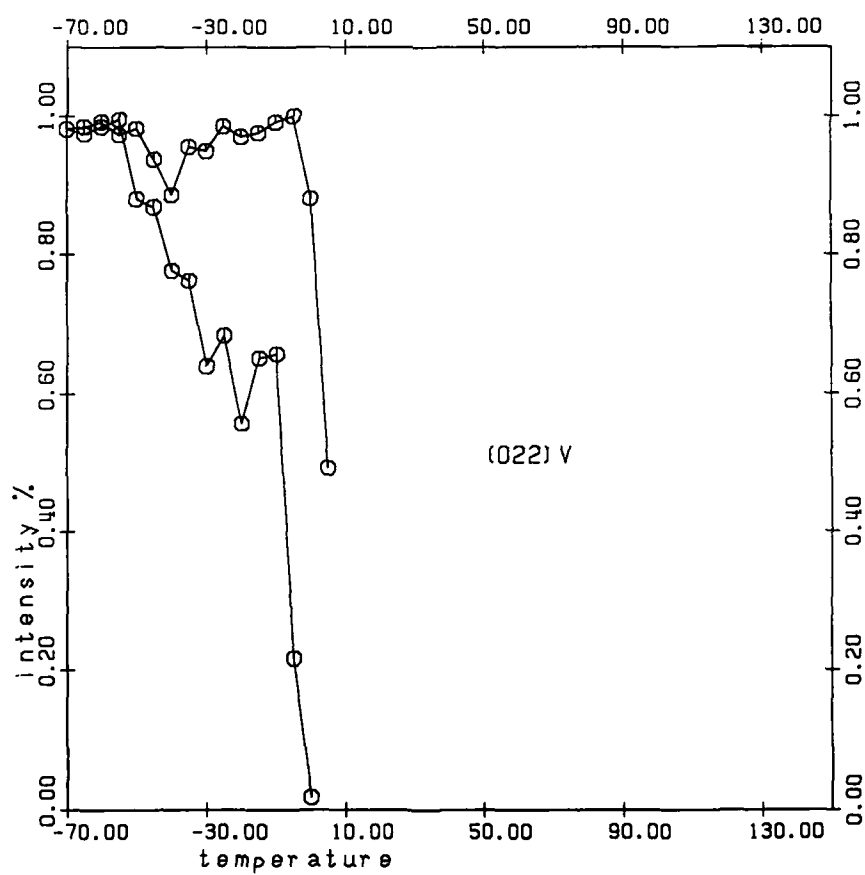
20/-70/140/-70°C



# Intensities

kf220988

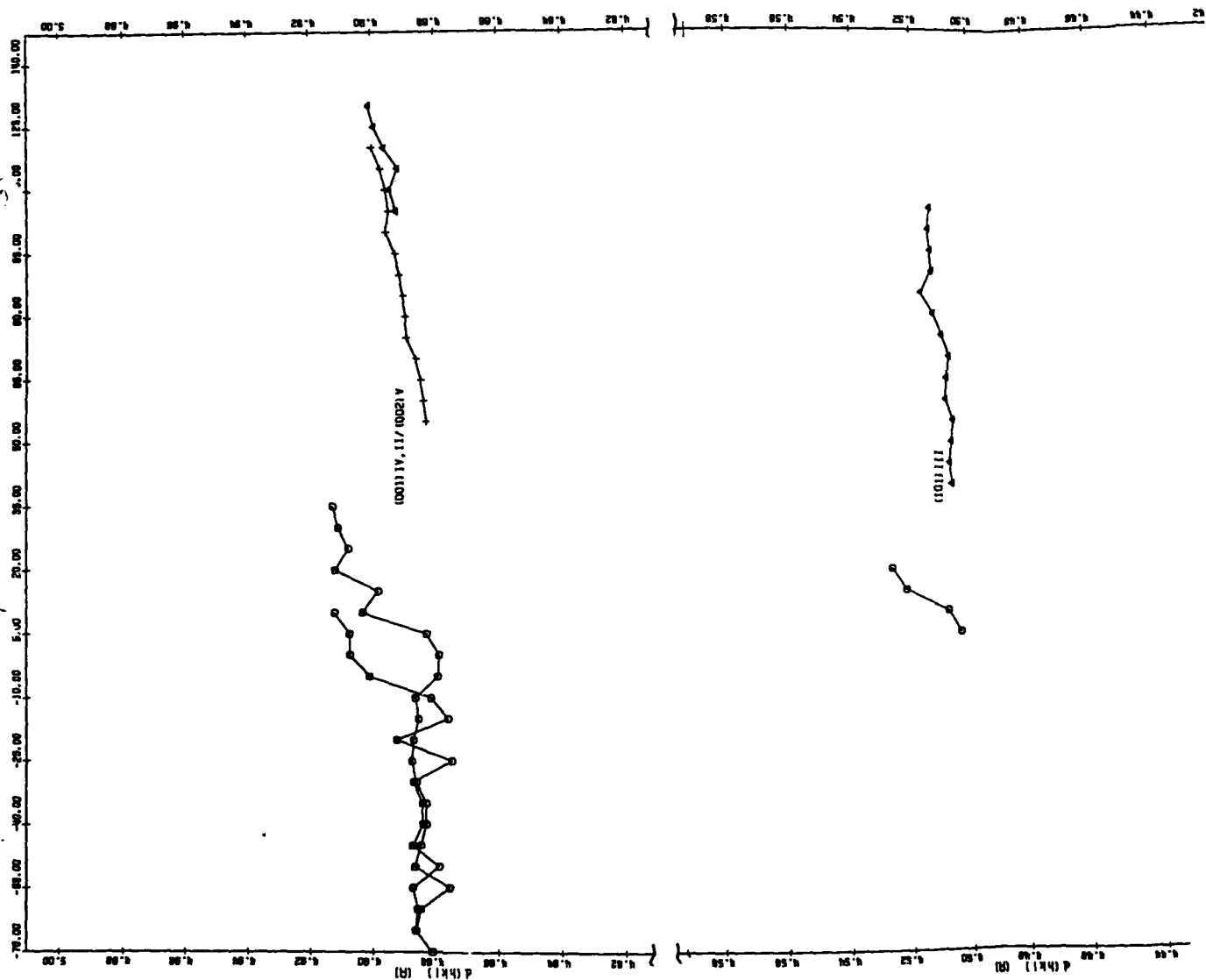
20/-70/140/-70°C



# Lattice Plane Distances

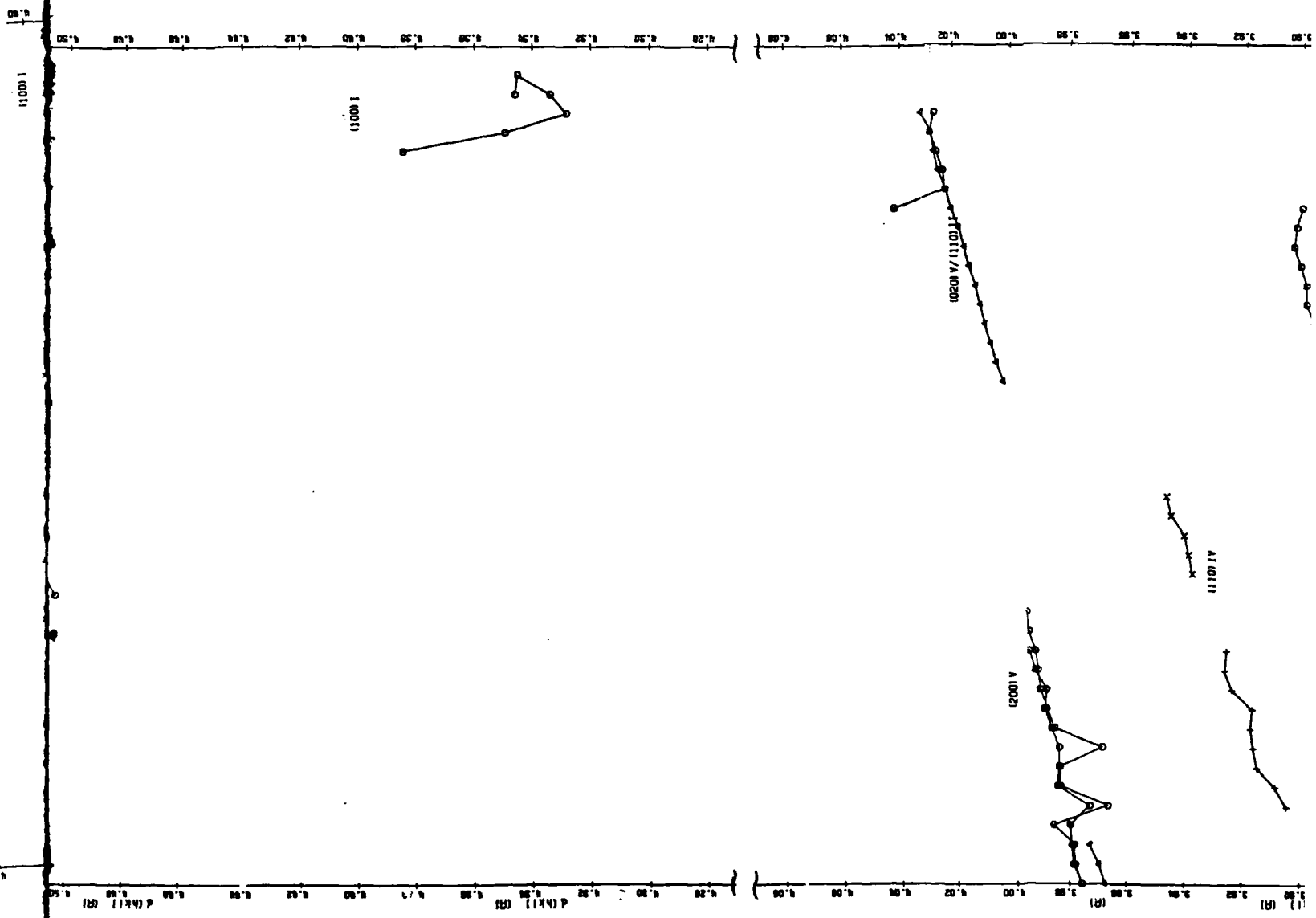
kf220988

20/-70/140/-70°C



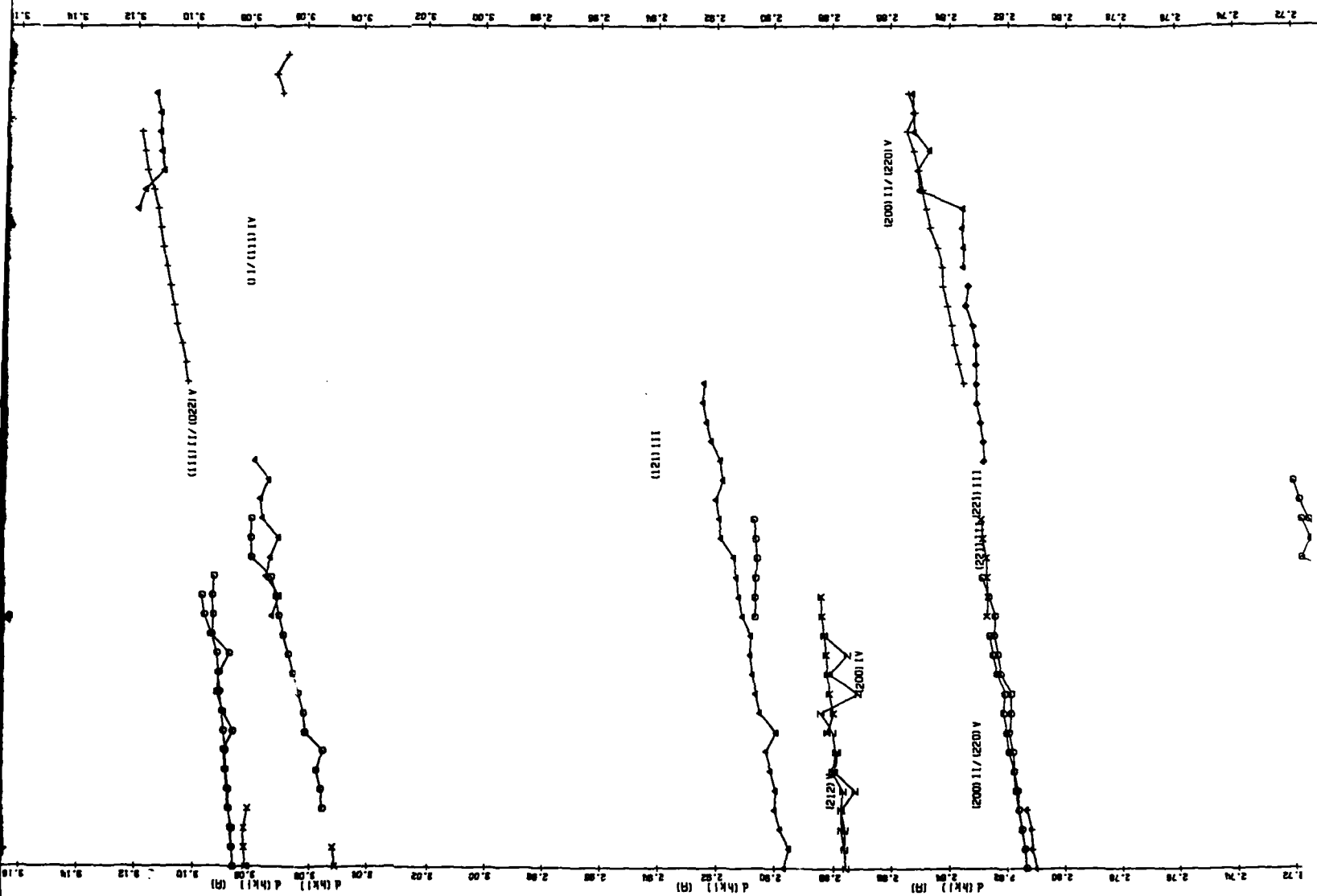
1075

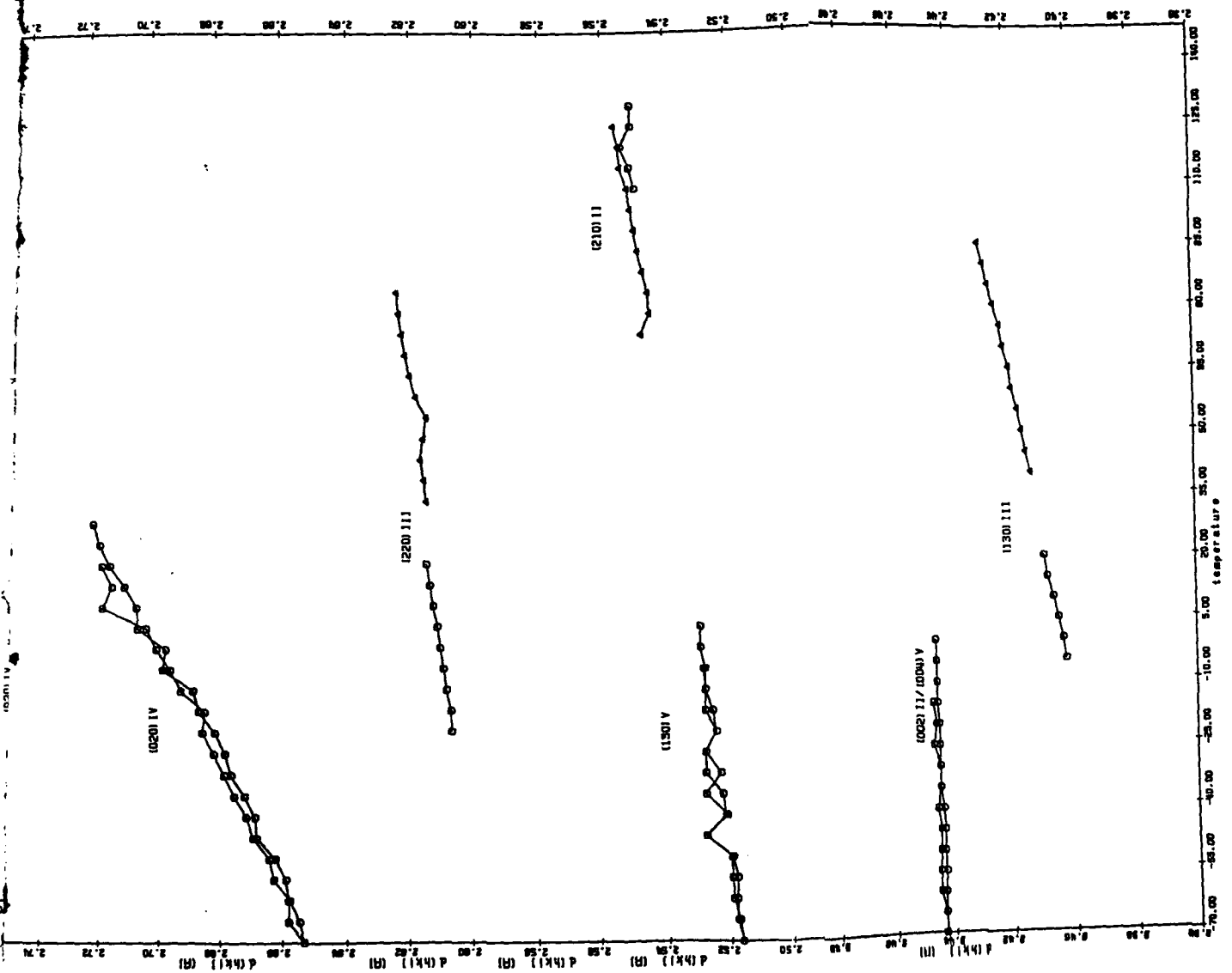




205





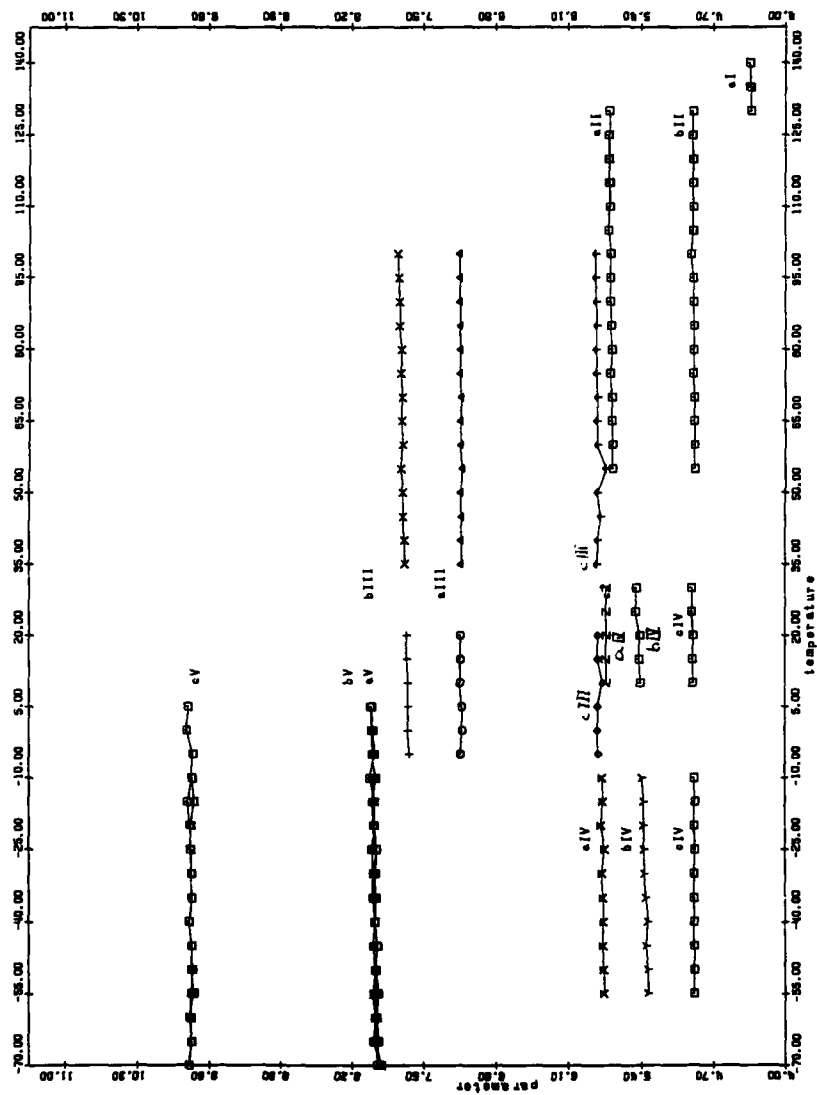


505

# Lattice Parameters

kf220988

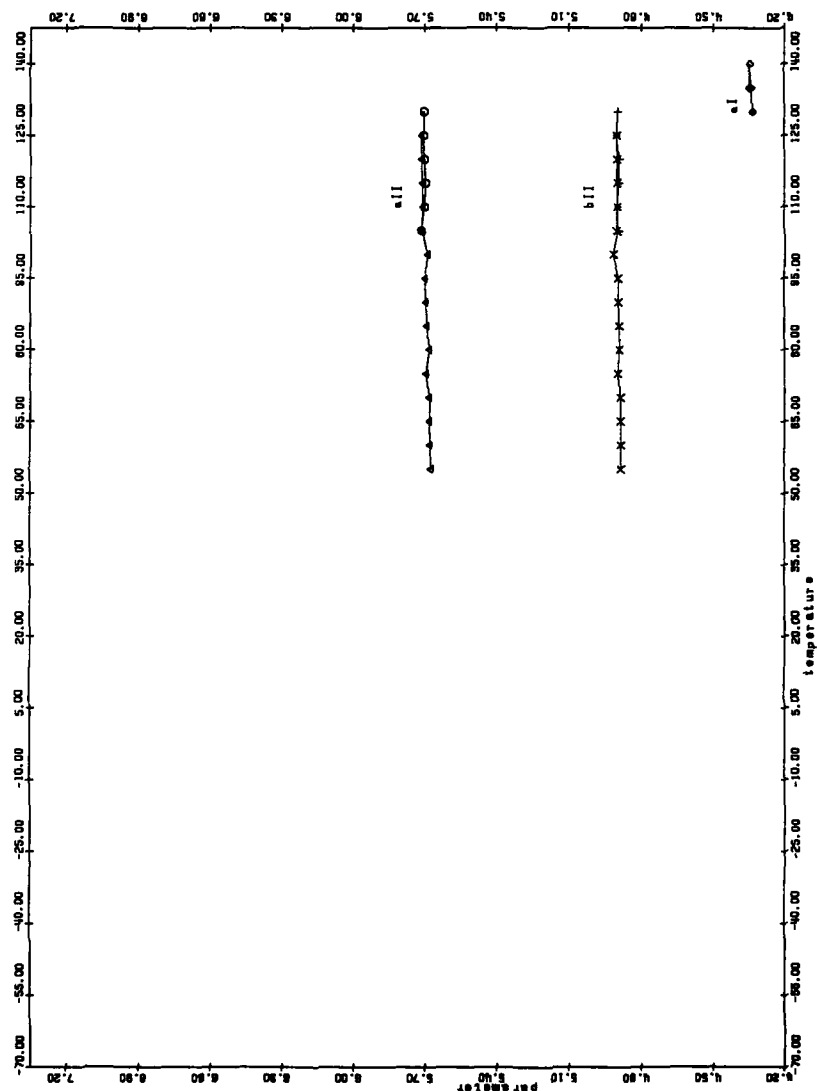
20/-70/140/-70°C



# Lattice Parameters

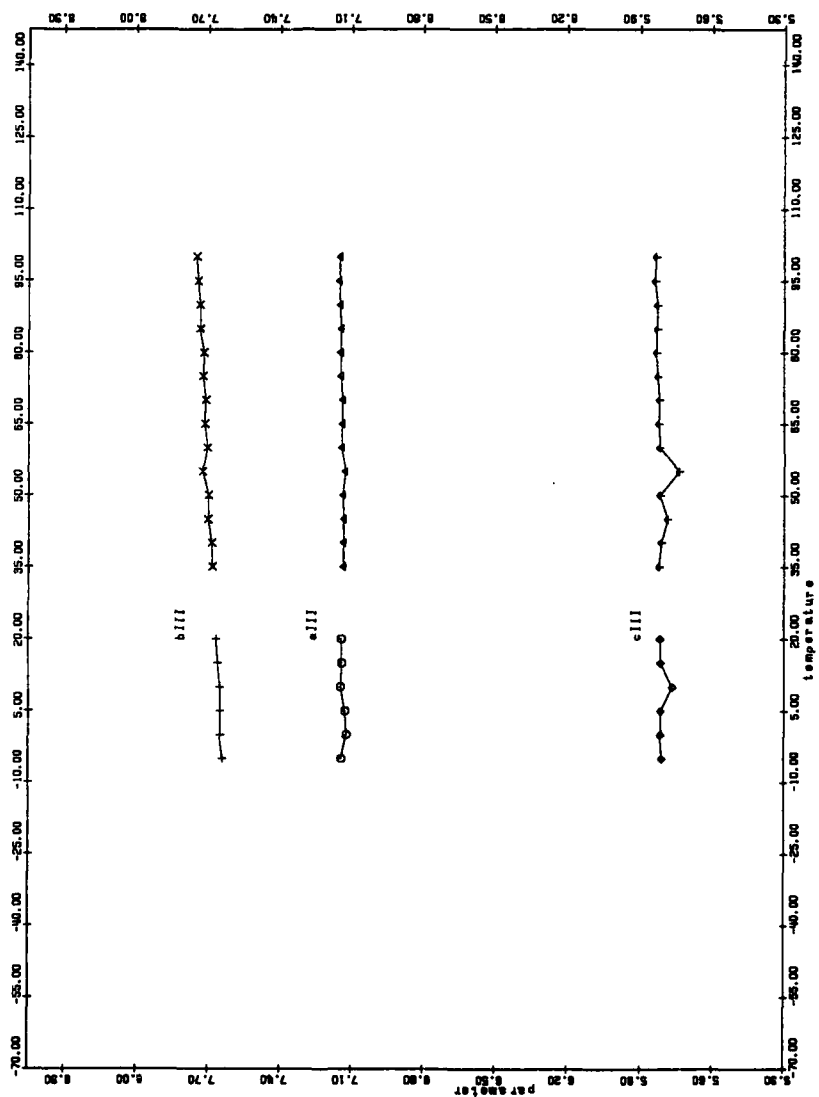
kf220988

20/-70/140/-70°C



# Lattice Parameters

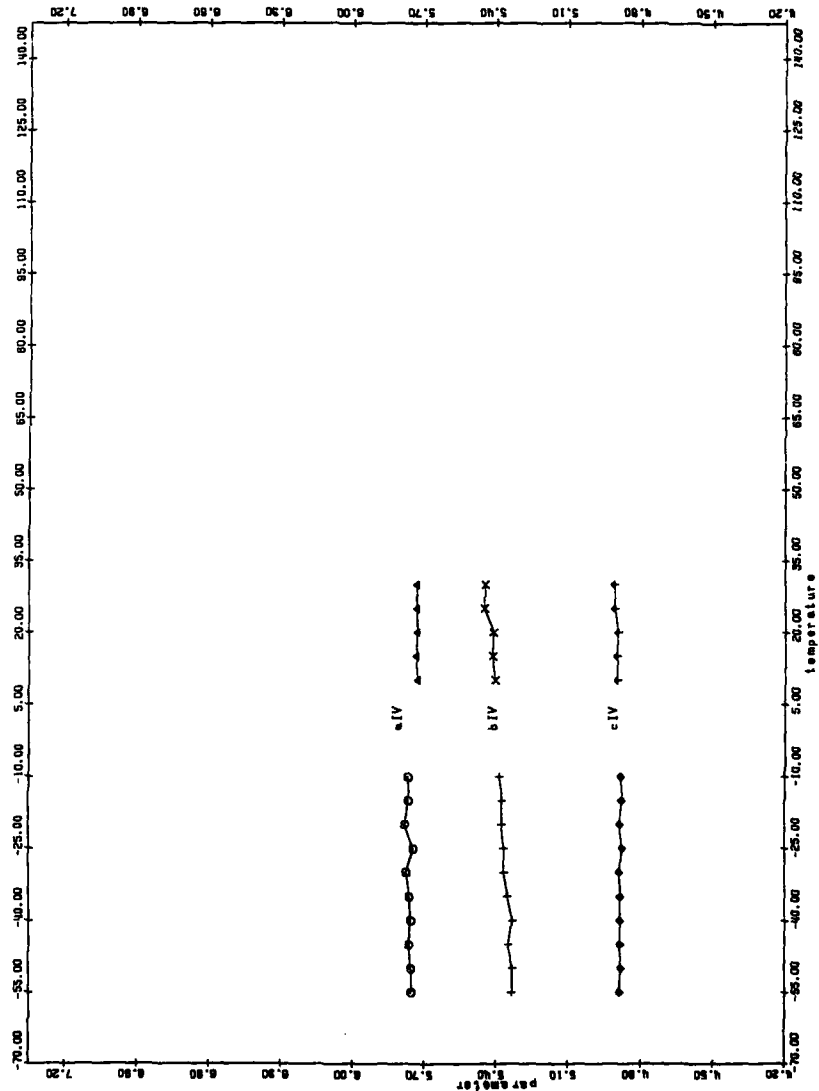
kf220988  
20/-70/140/-70°C



# Lattice Parameters

kf220988

20/-70/140/-70°C

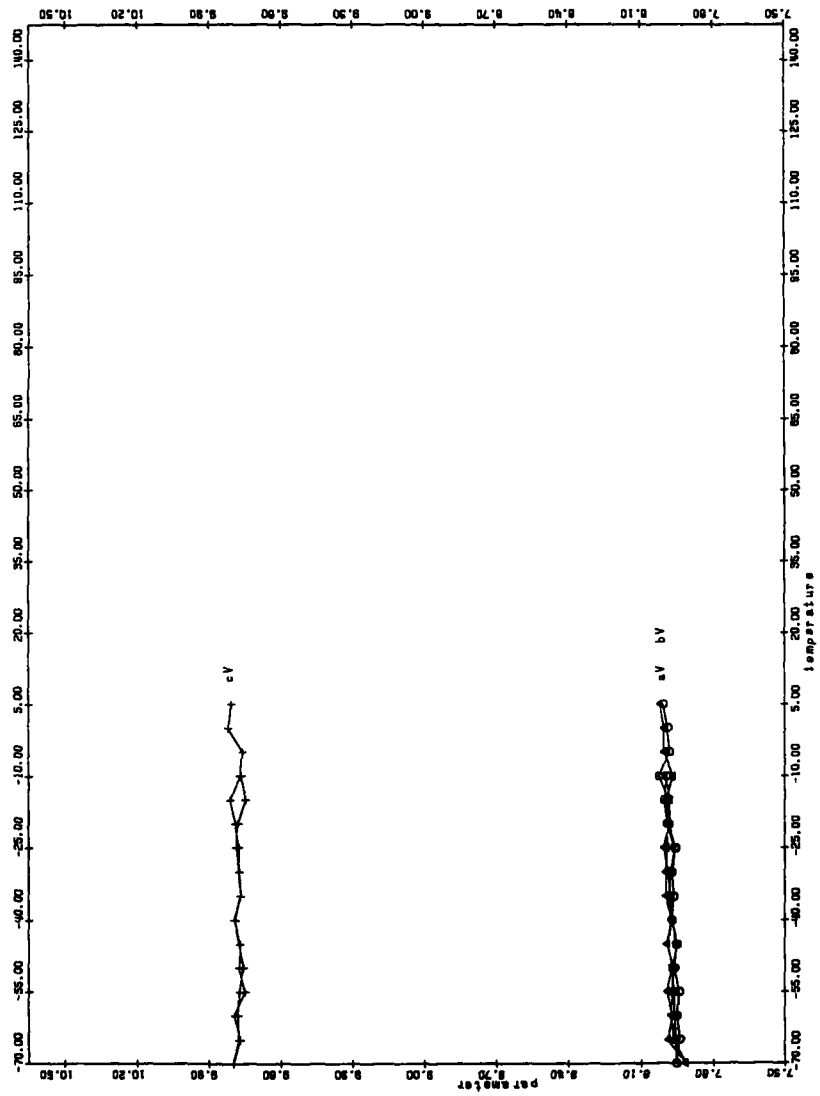




# Lattice Parameters

kf220988

20/-70/140/-70°C



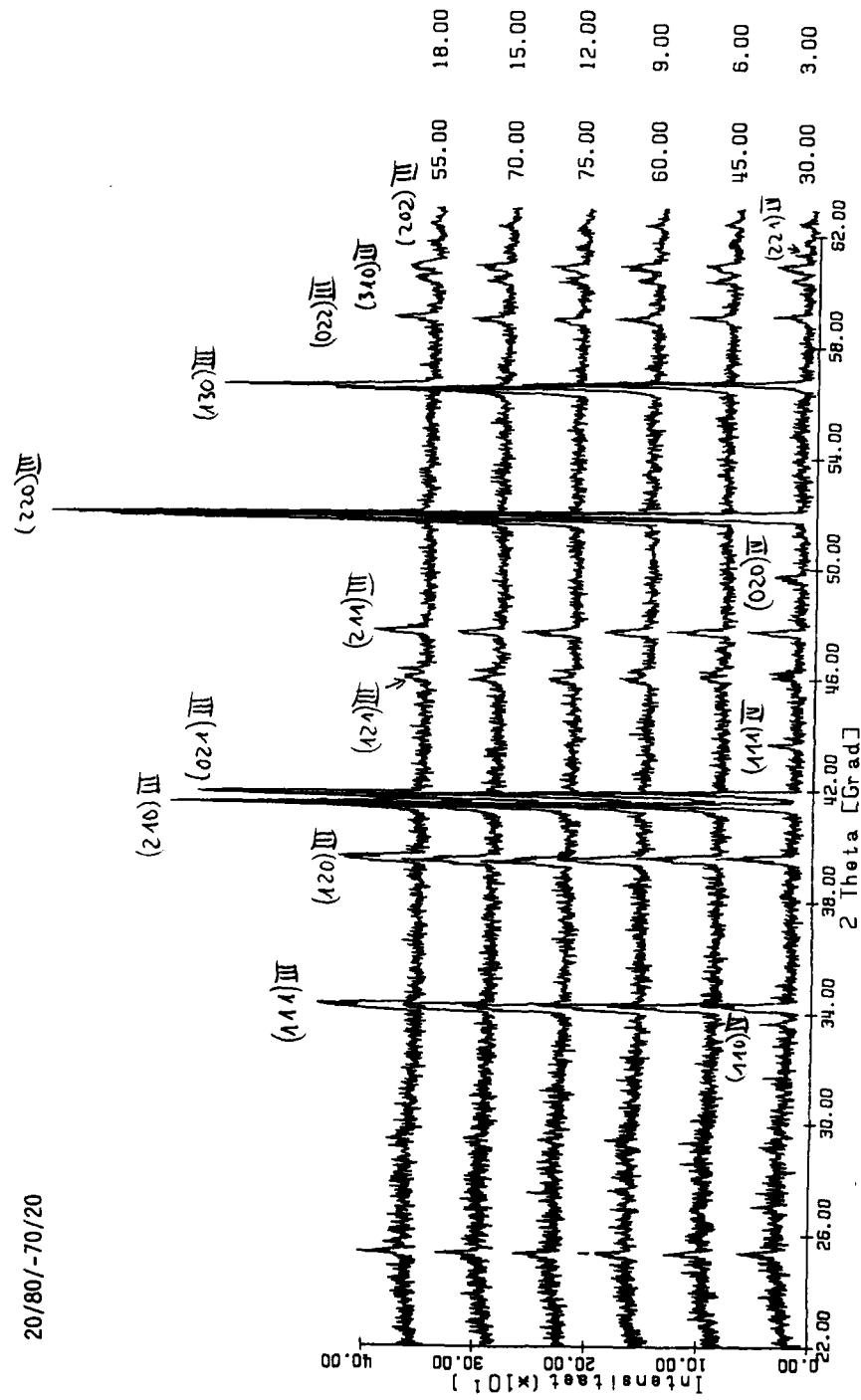
*Series*  
*KF 280988*  
*humide*

*Temperature*  
*Program*  
*20/80/-70/80*

# Diffraction Patterns

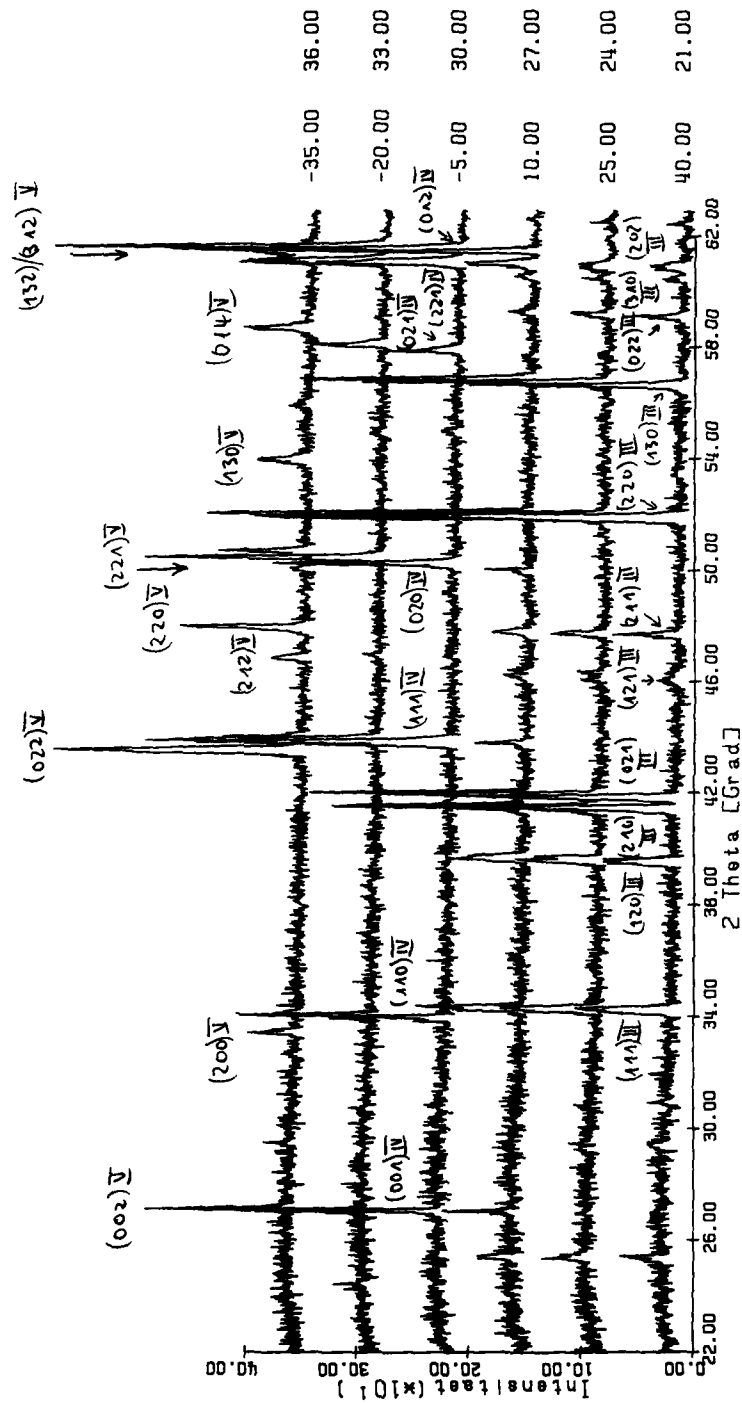
kf280988

20/80/-70/20



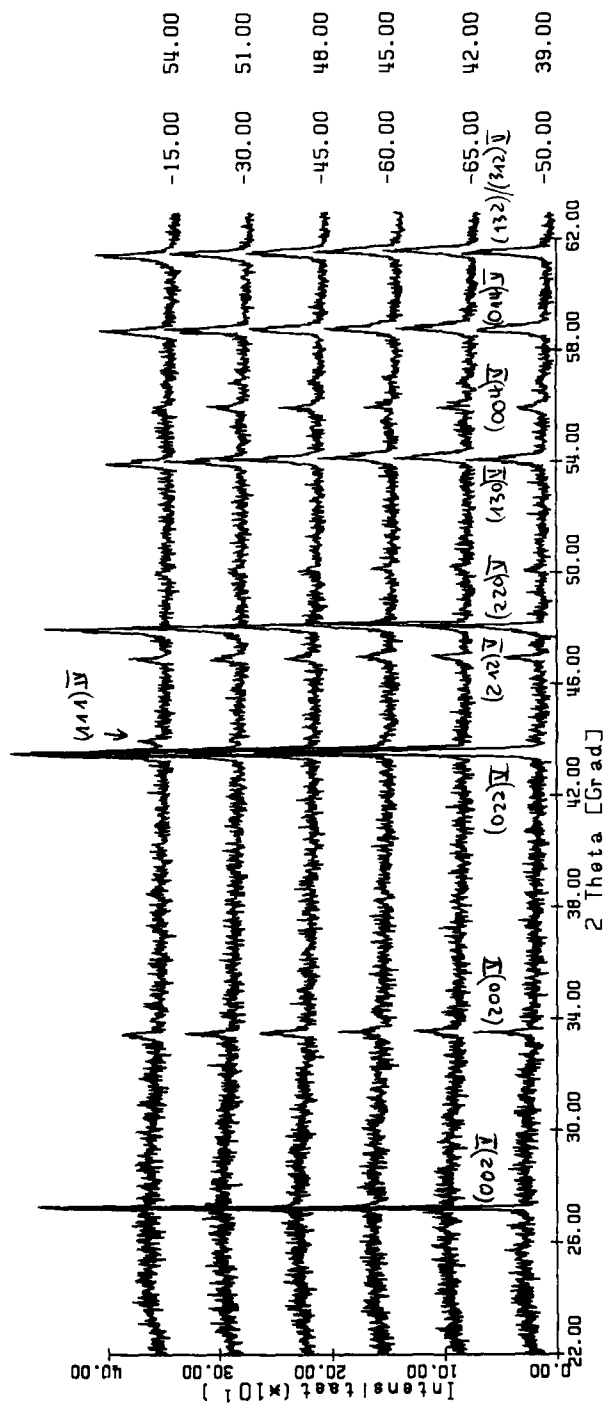
kf280988

20/80/-70/20



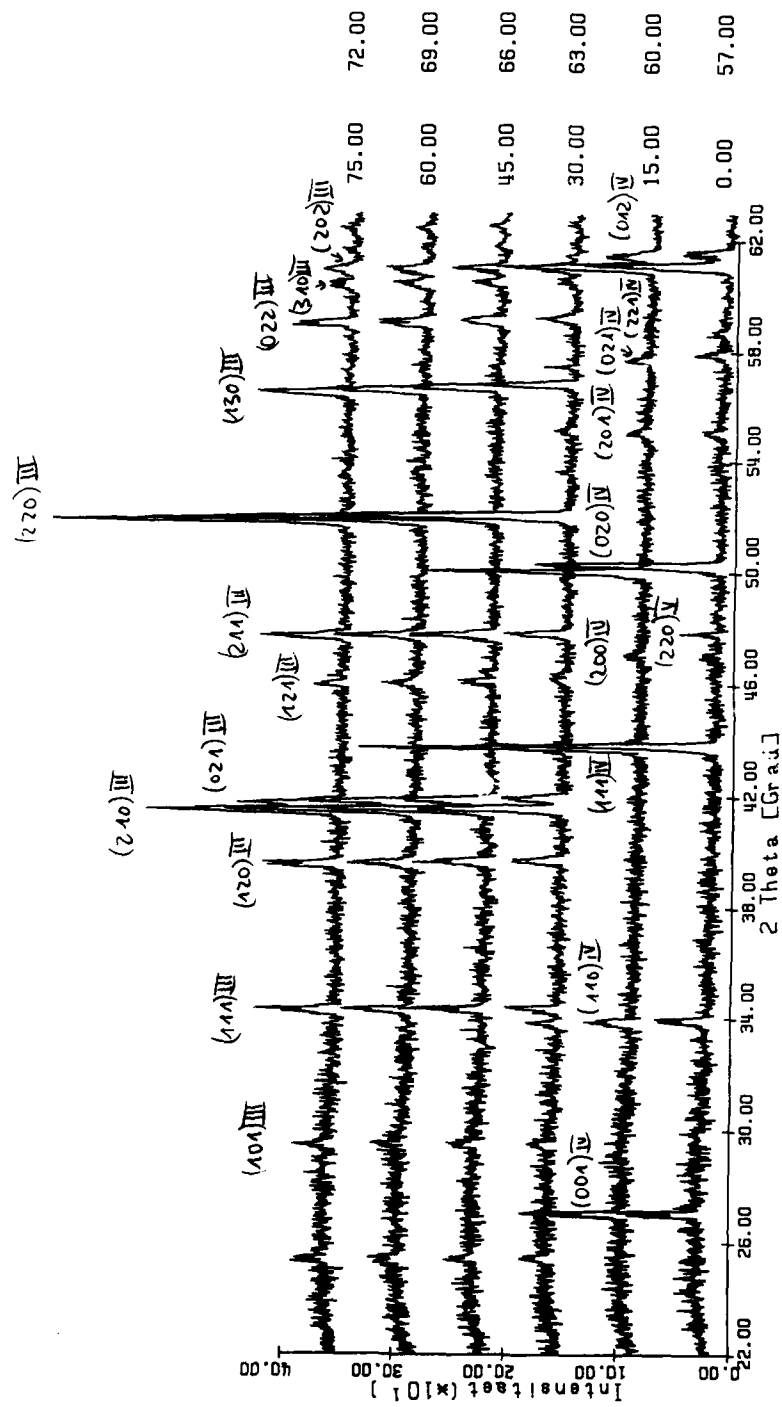
kf280988

20/80/-70/20



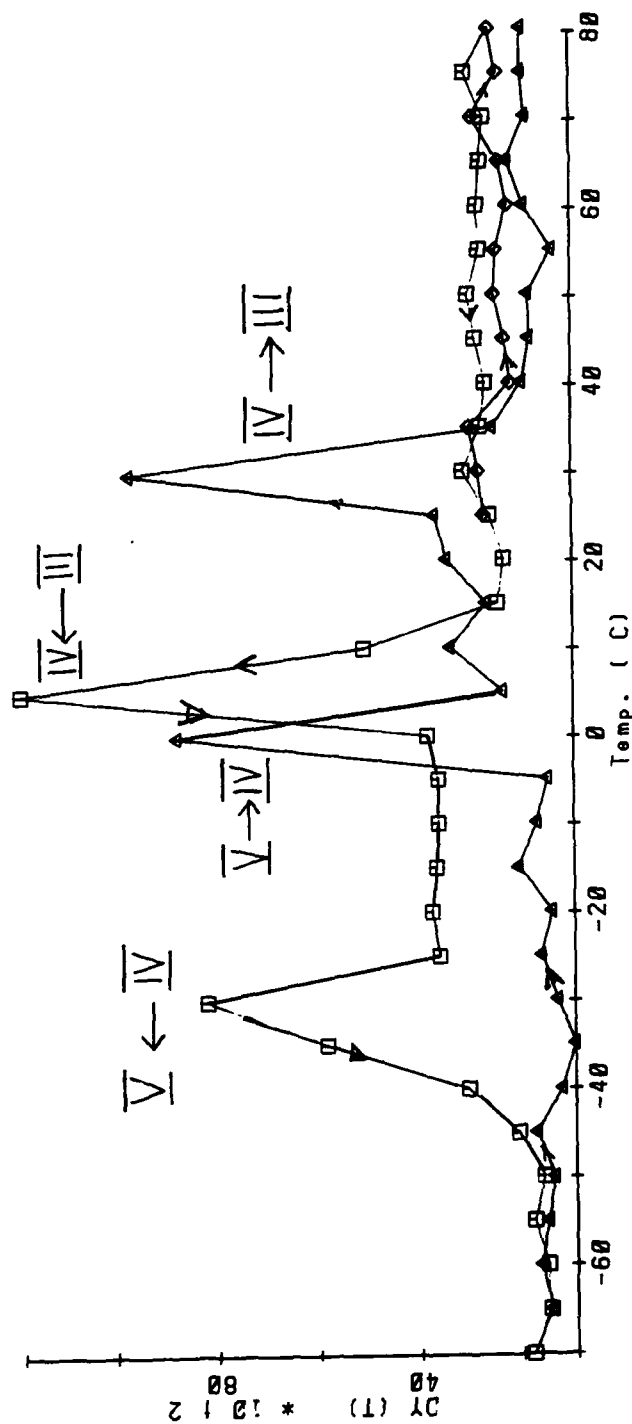
kf280988

20/80/-70/20



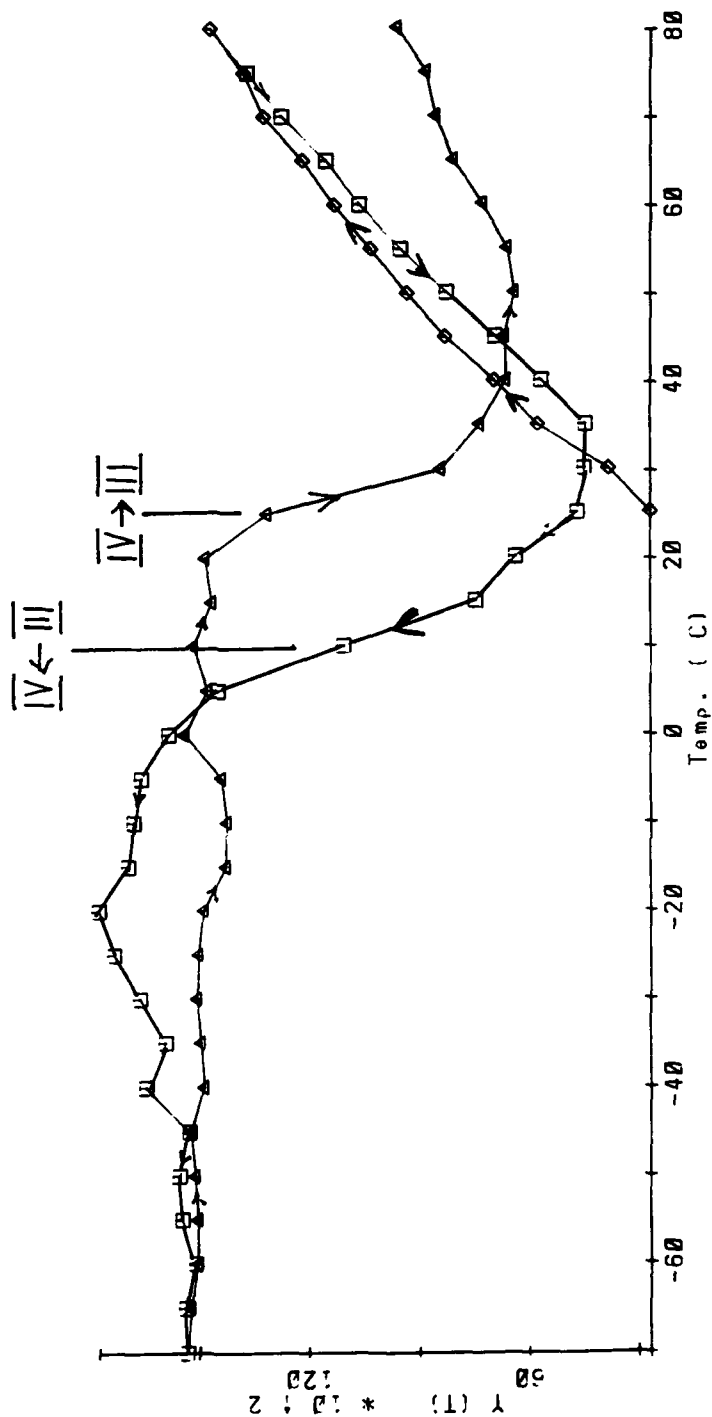
# Difference Curve $\Delta y(T)$

2% KF hum  
kf280988  
20/80/-70/20



# Difference Curve Y(T)

2% KF hum  
kf280988  
20/80/-70/20

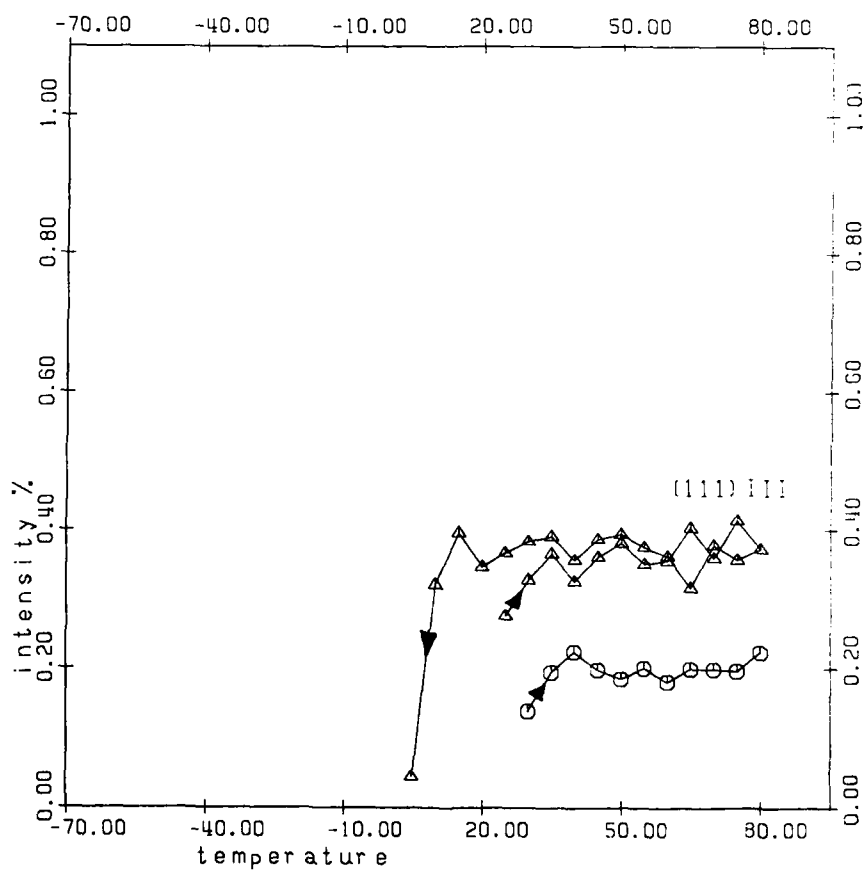




# Intensities

kf280988

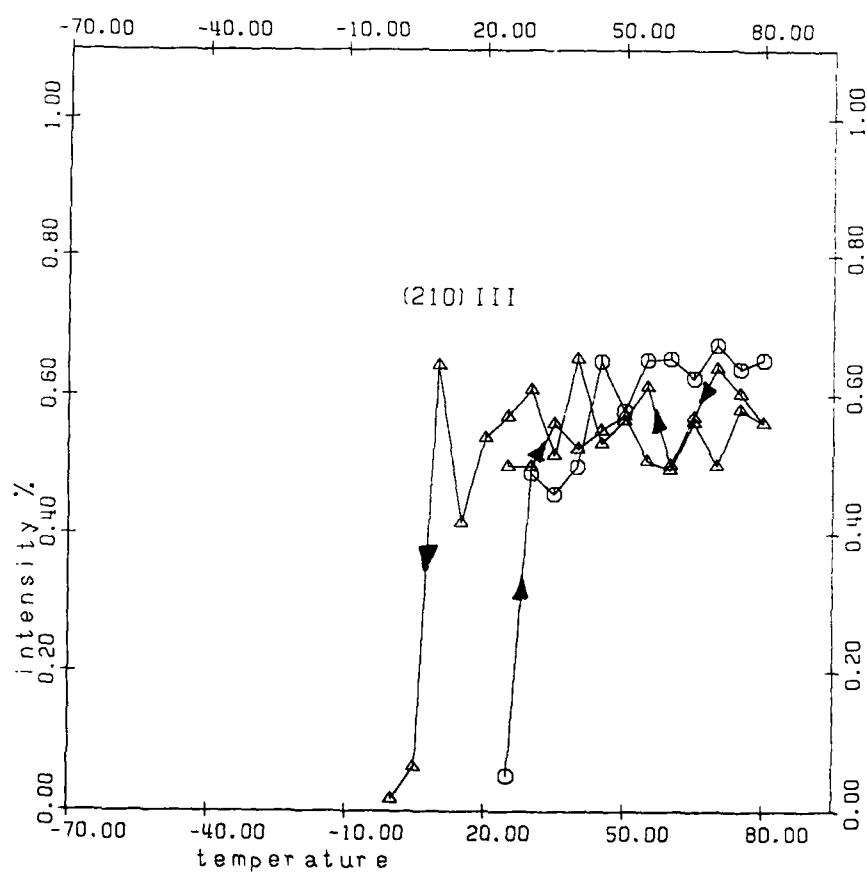
2% KF hum 20/80/-70/80



# Intensities

kf280988

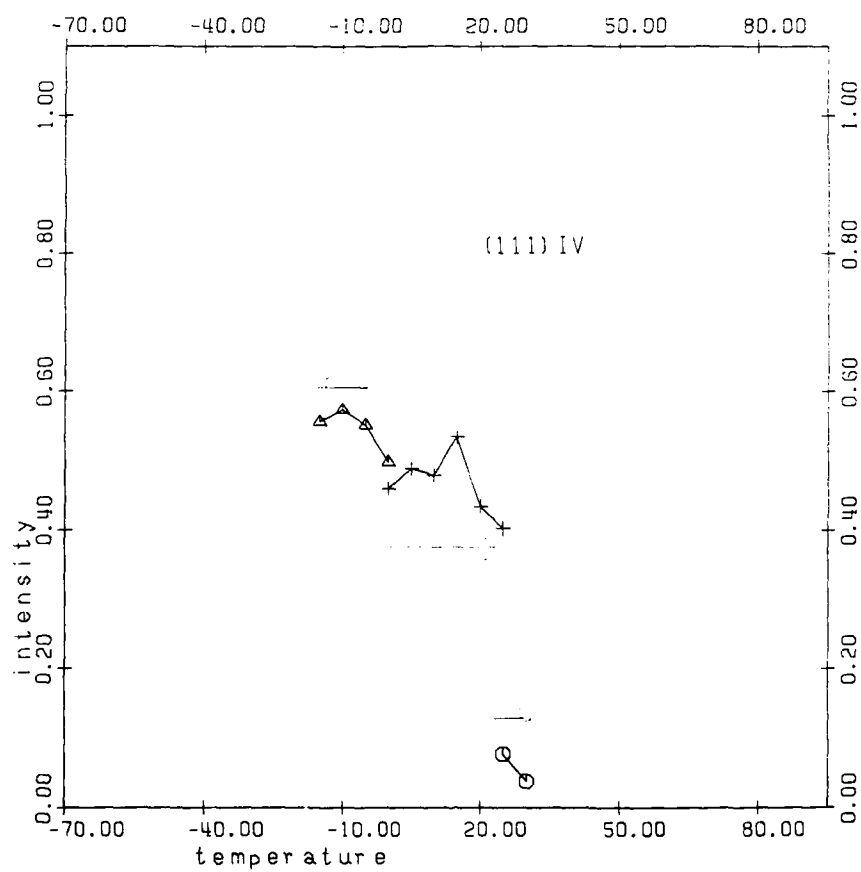
2% KF hum 20/80/-70/80



# Intensities

kf280988

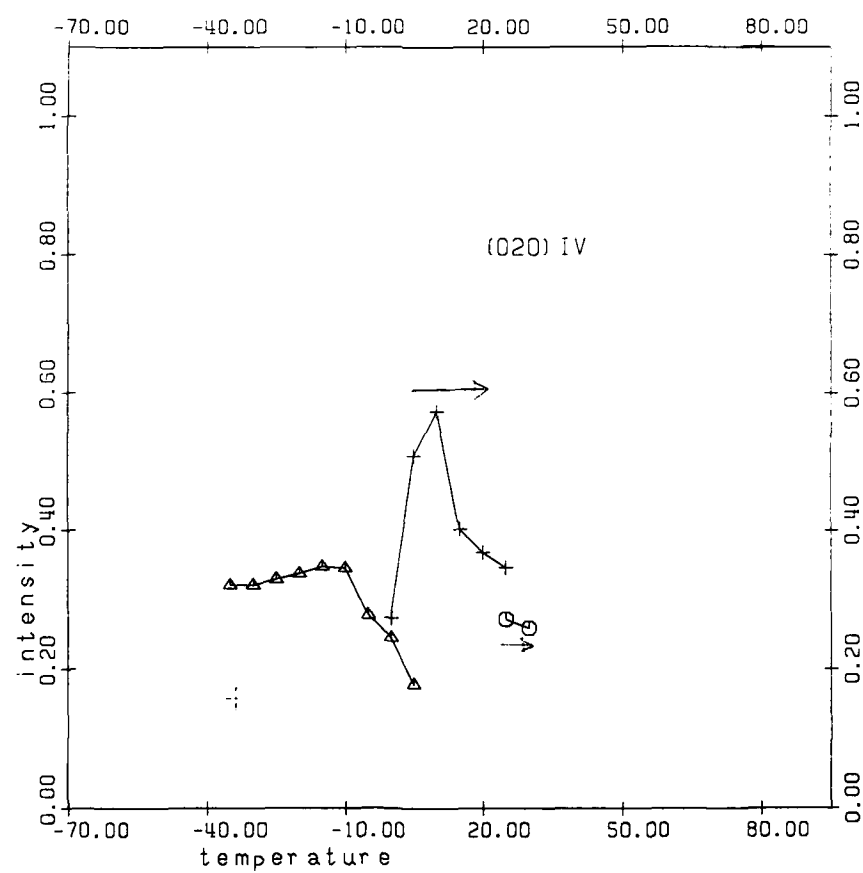
20/80/-70/20



# Intensities

kf280988

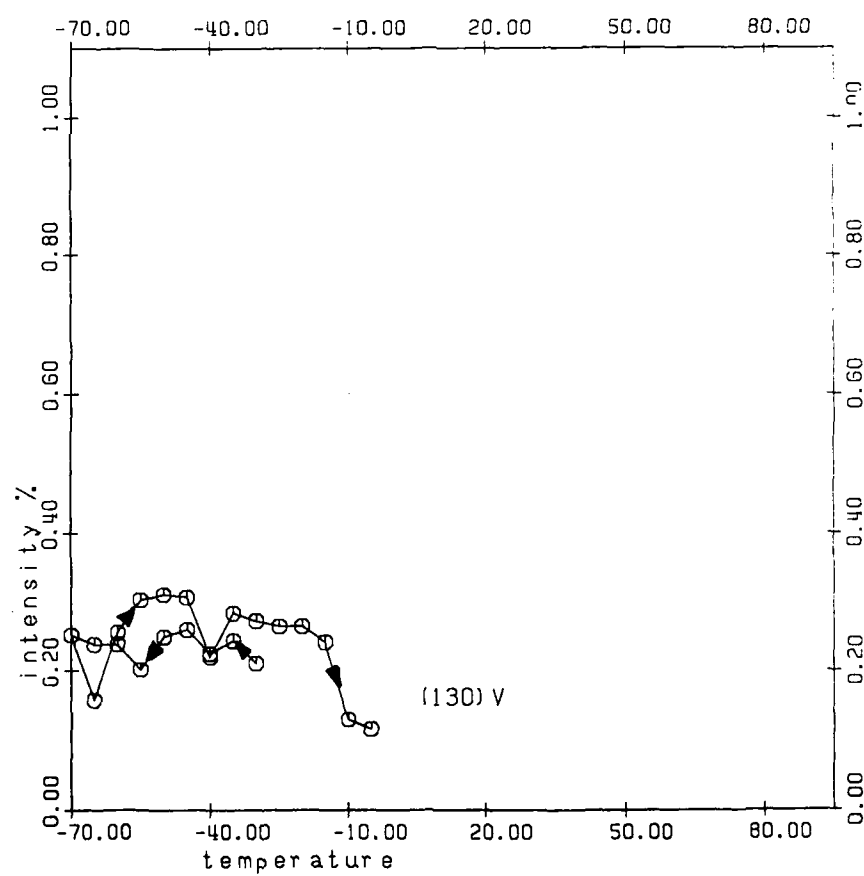
20/80/-70/20



# Intensities

kf280988

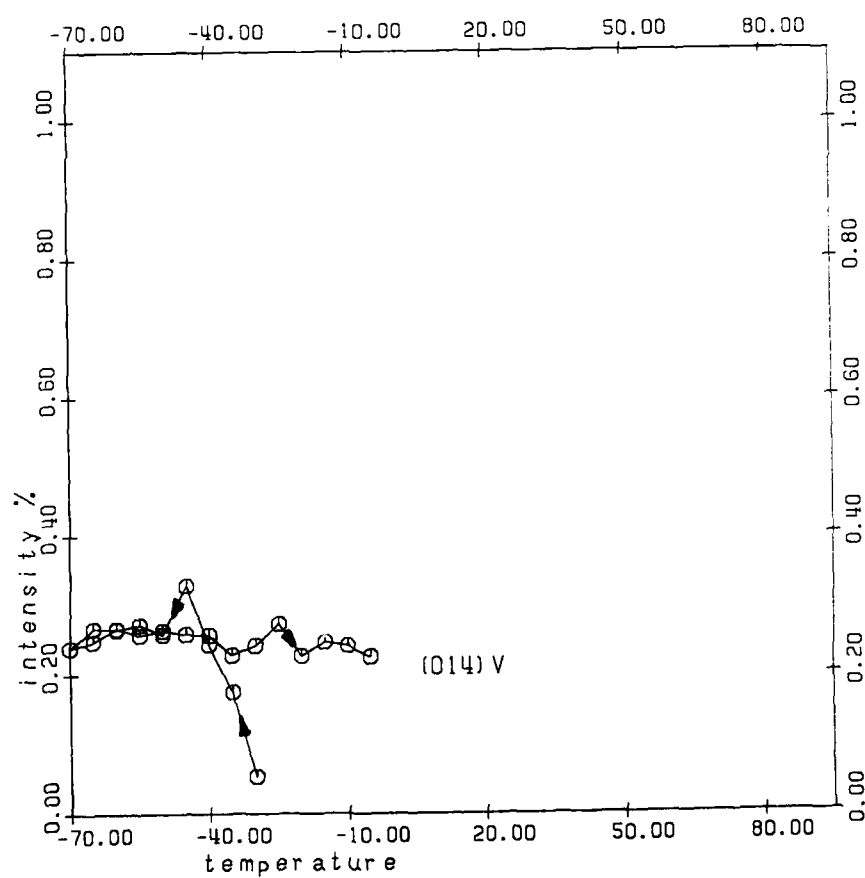
2% KF hum 20/80/-70/80



# Intensities

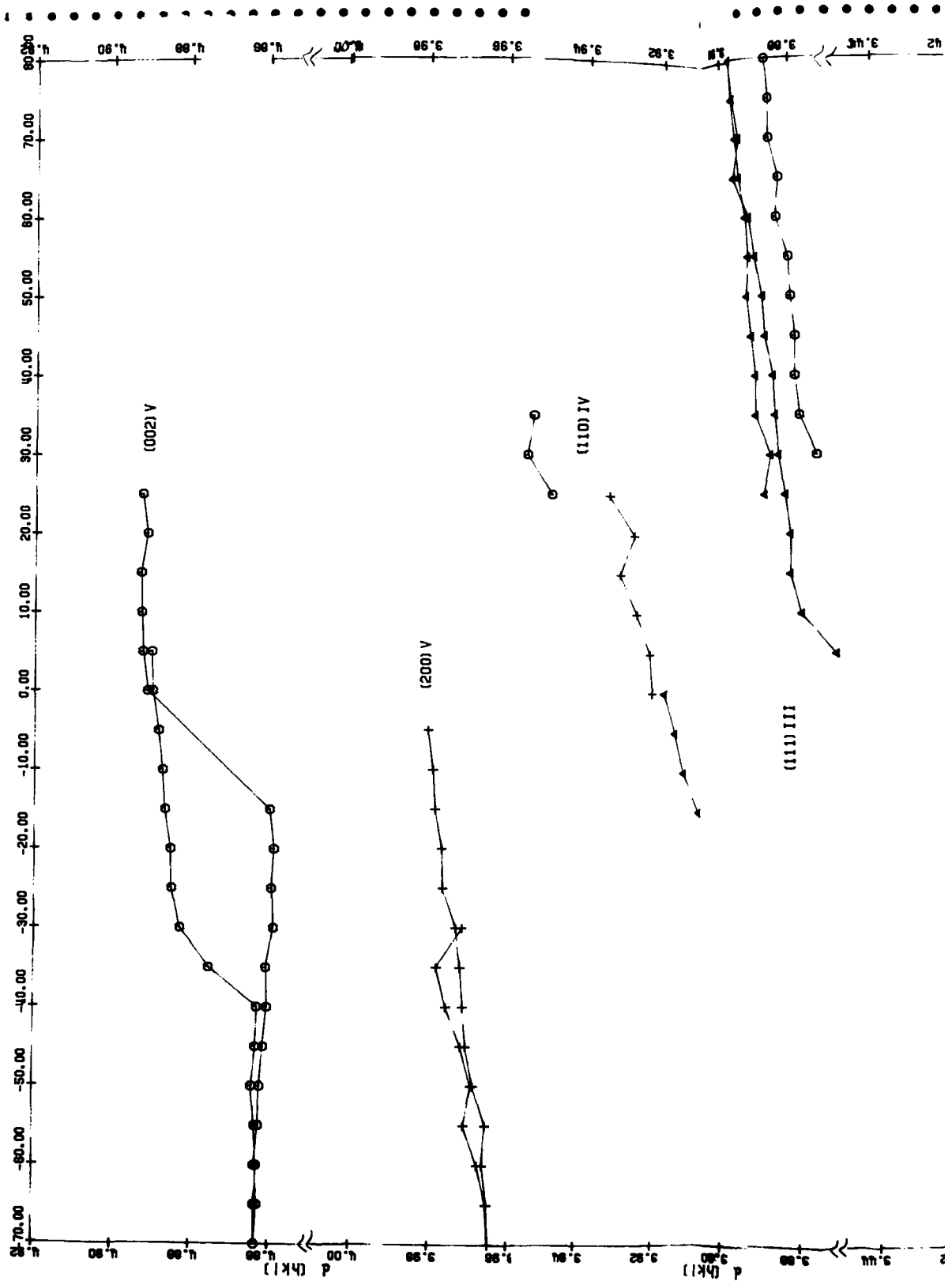
kf280988

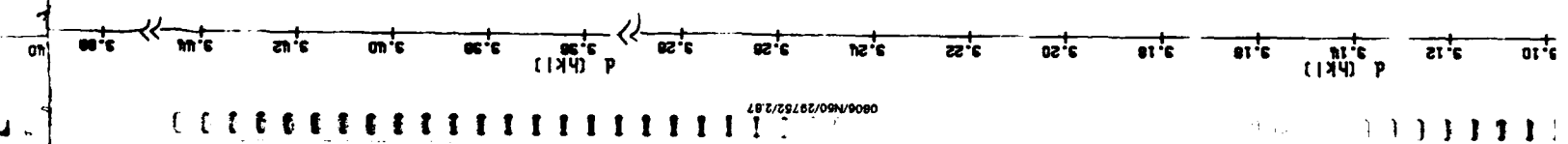
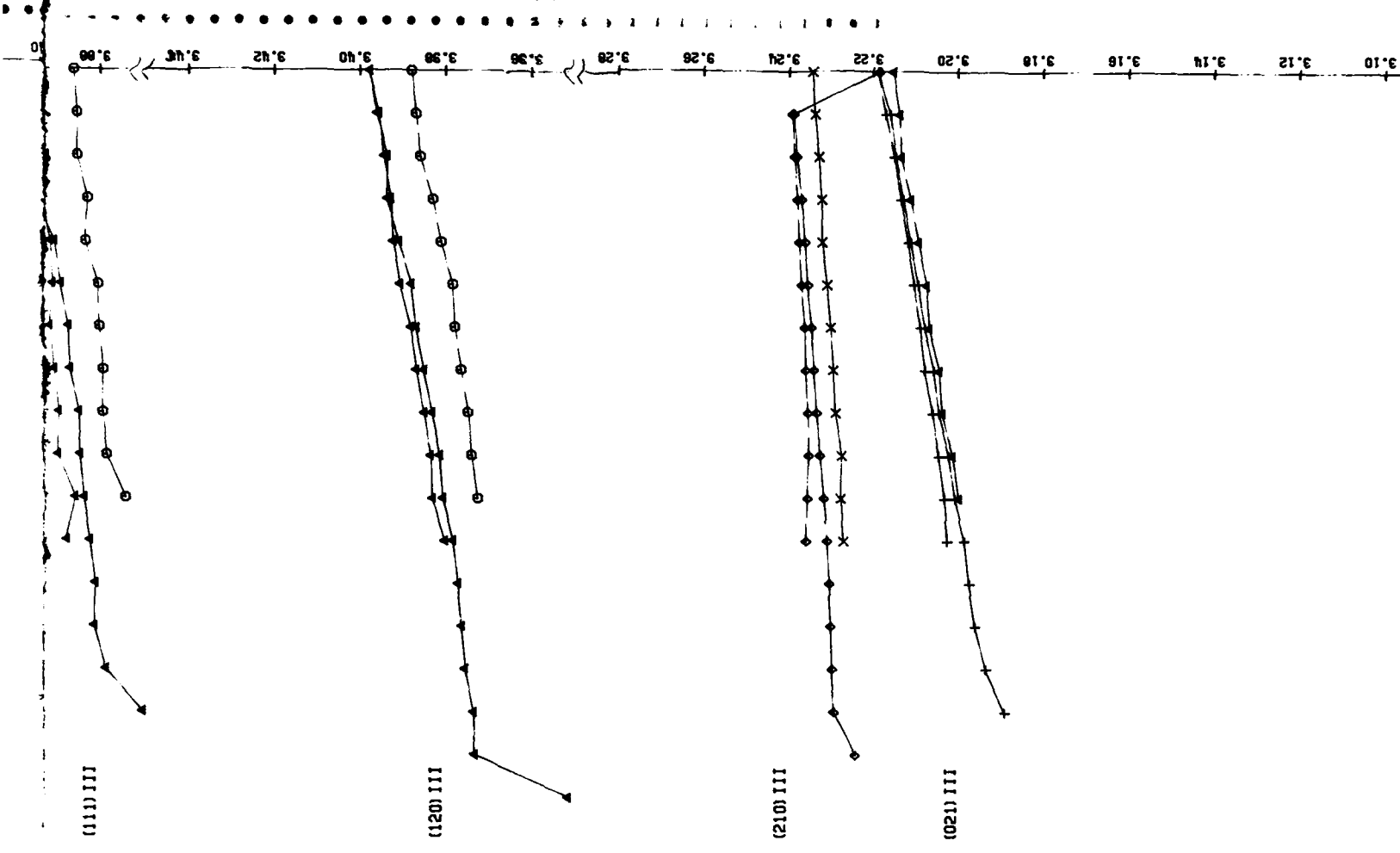
2% KF hum 20/80/-70/80



# Lattice Plane Distances

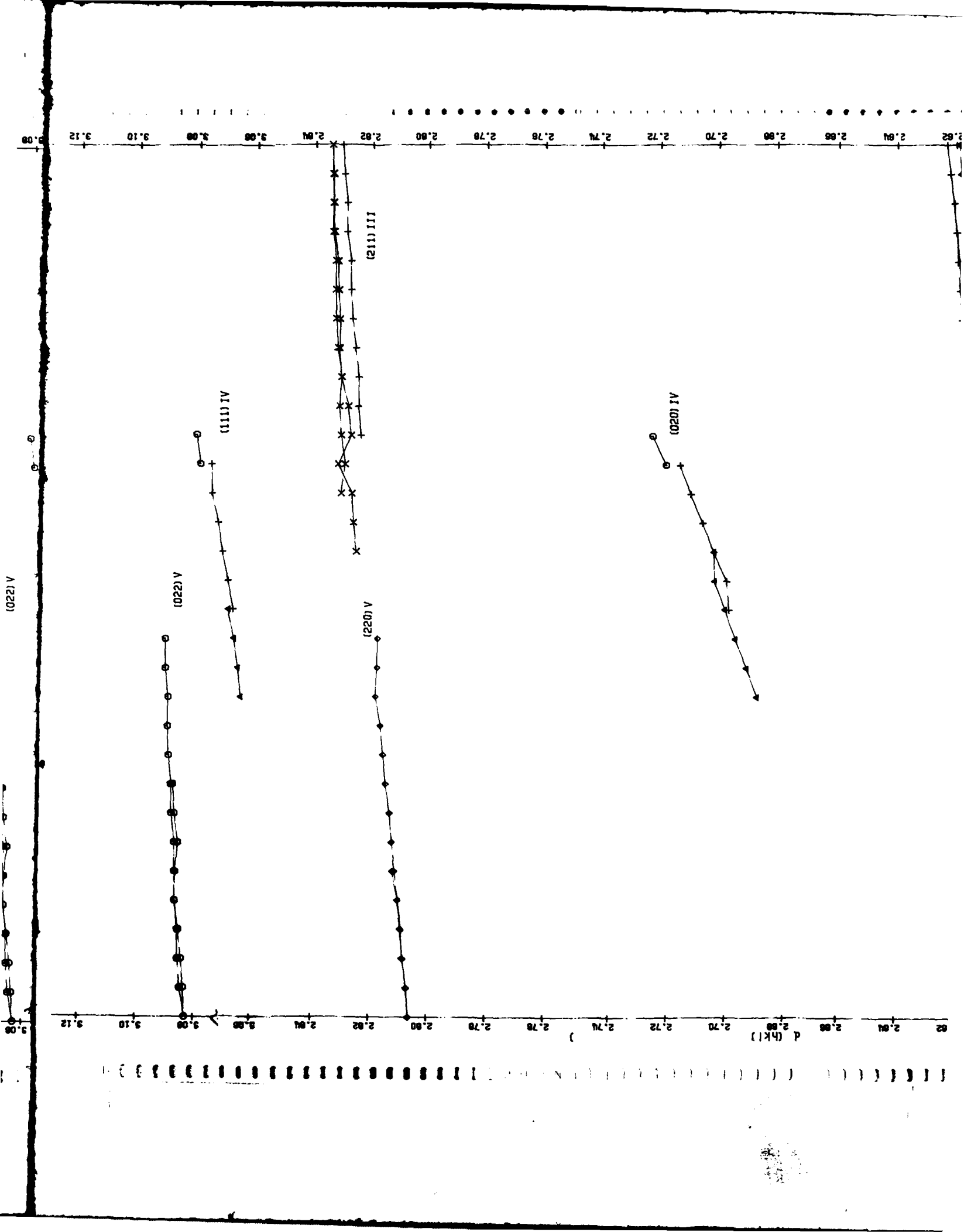
kf280988  
20/80/-70/20

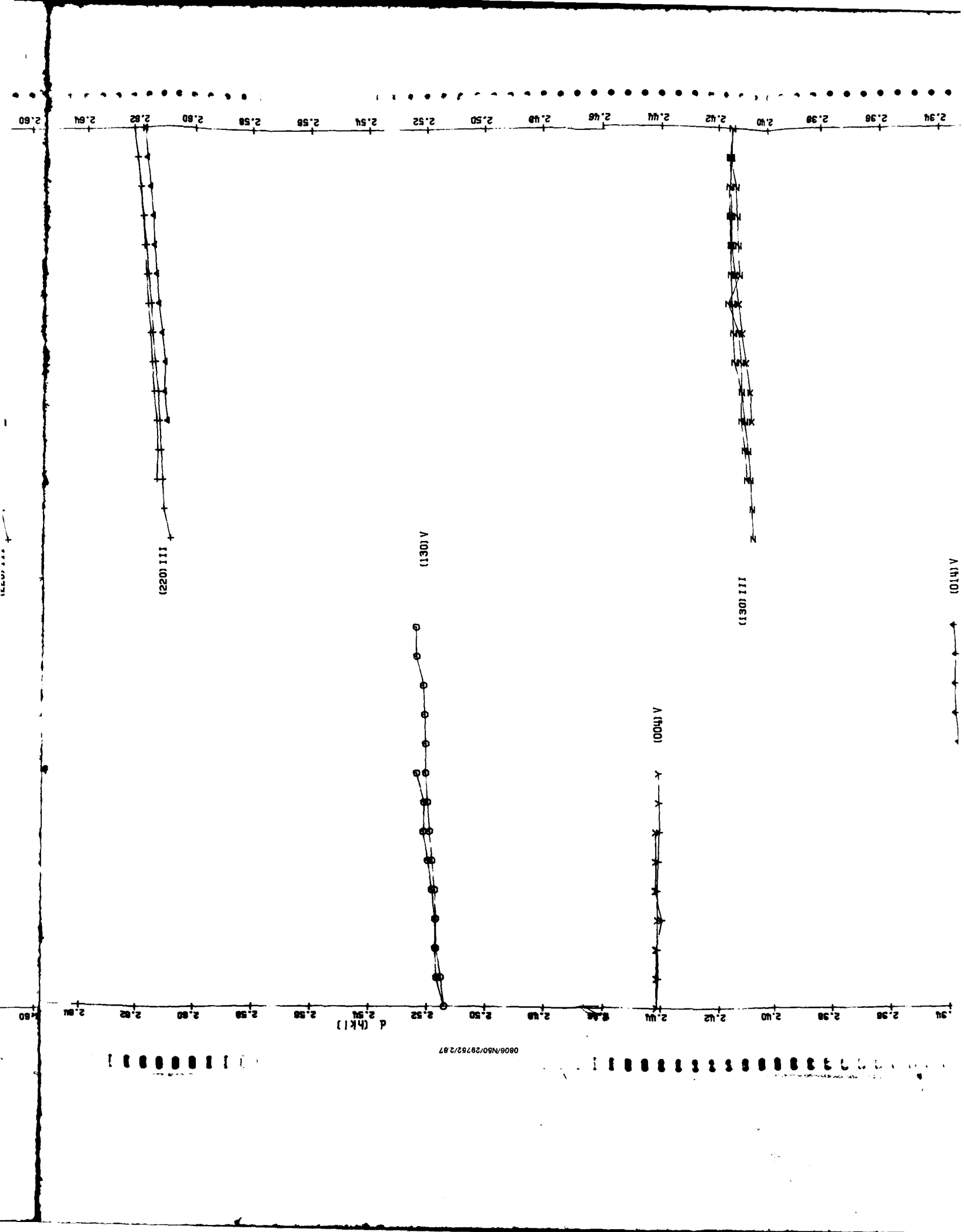


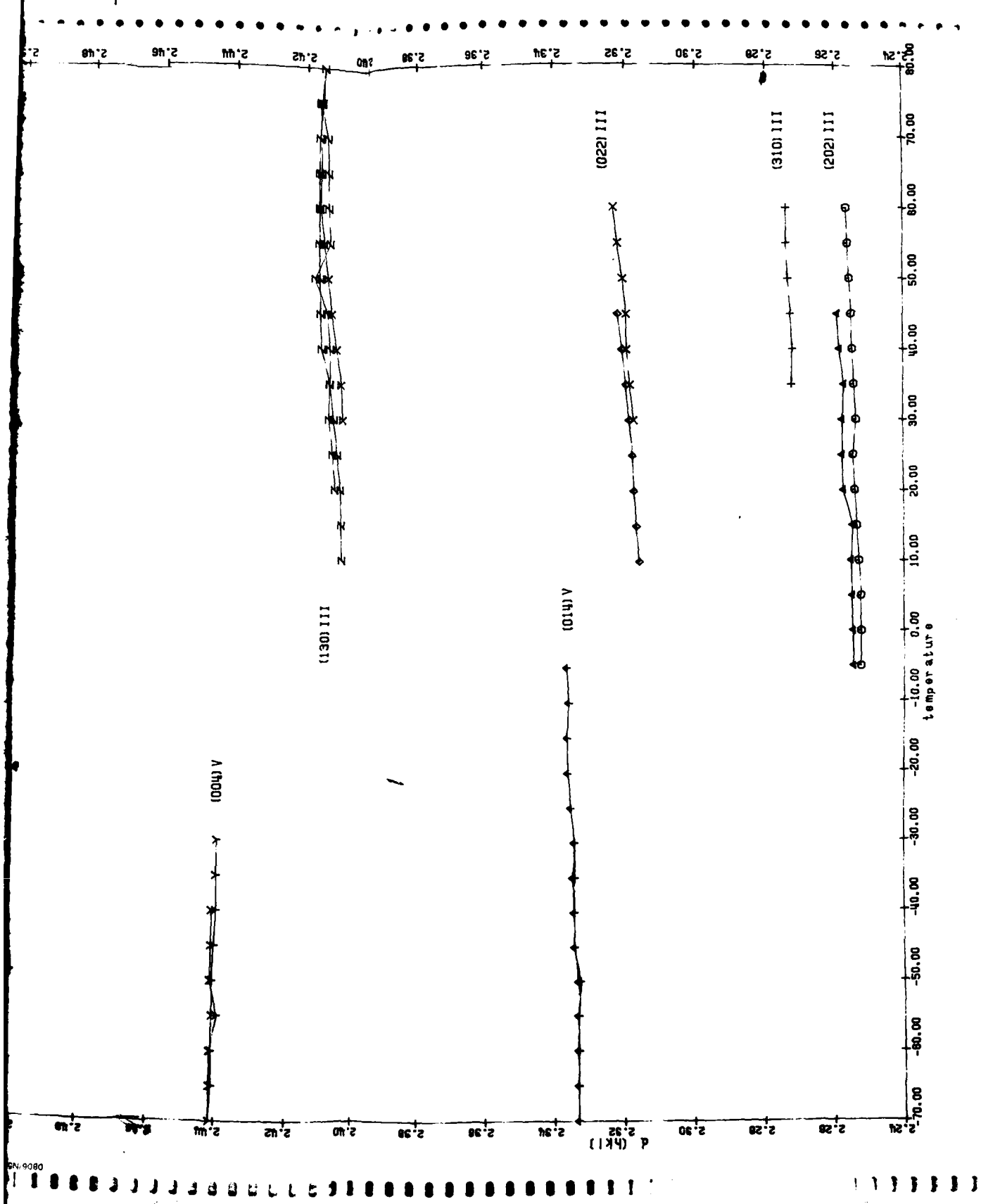


0806/460/28752/2.87





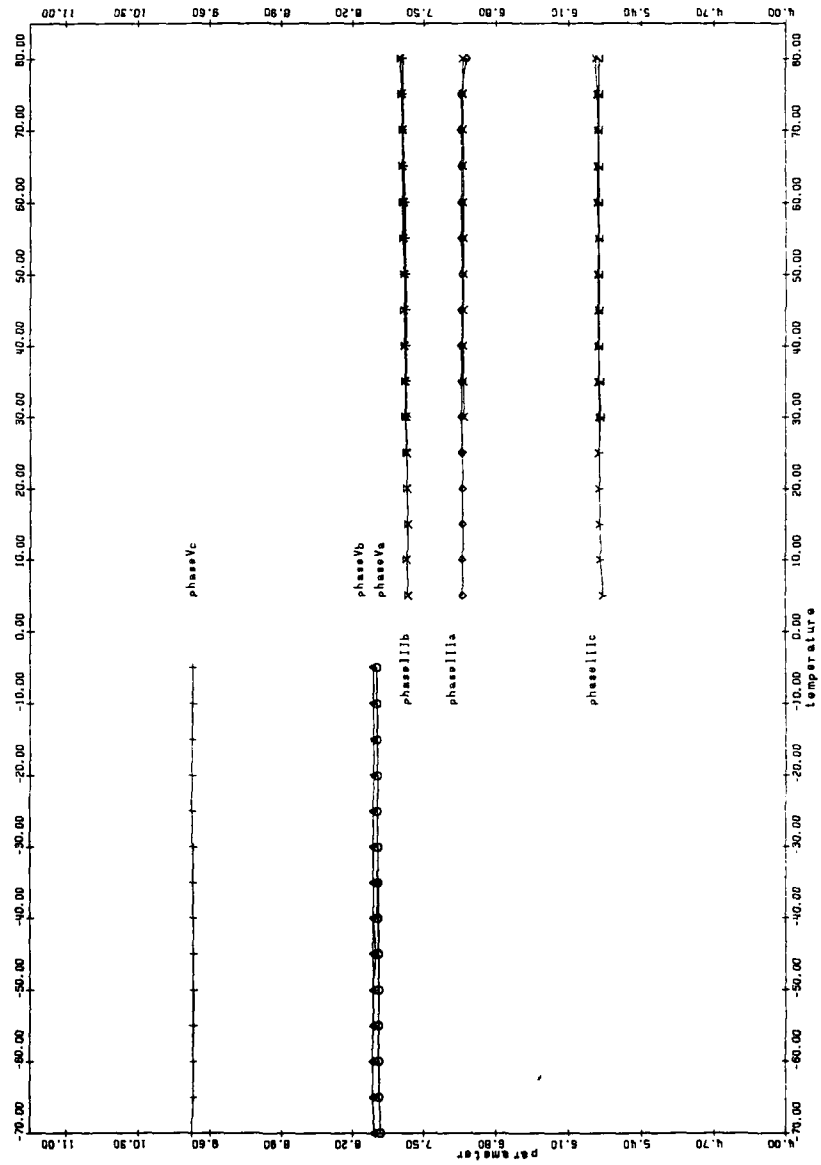




# Lattice Parameters

kf280988

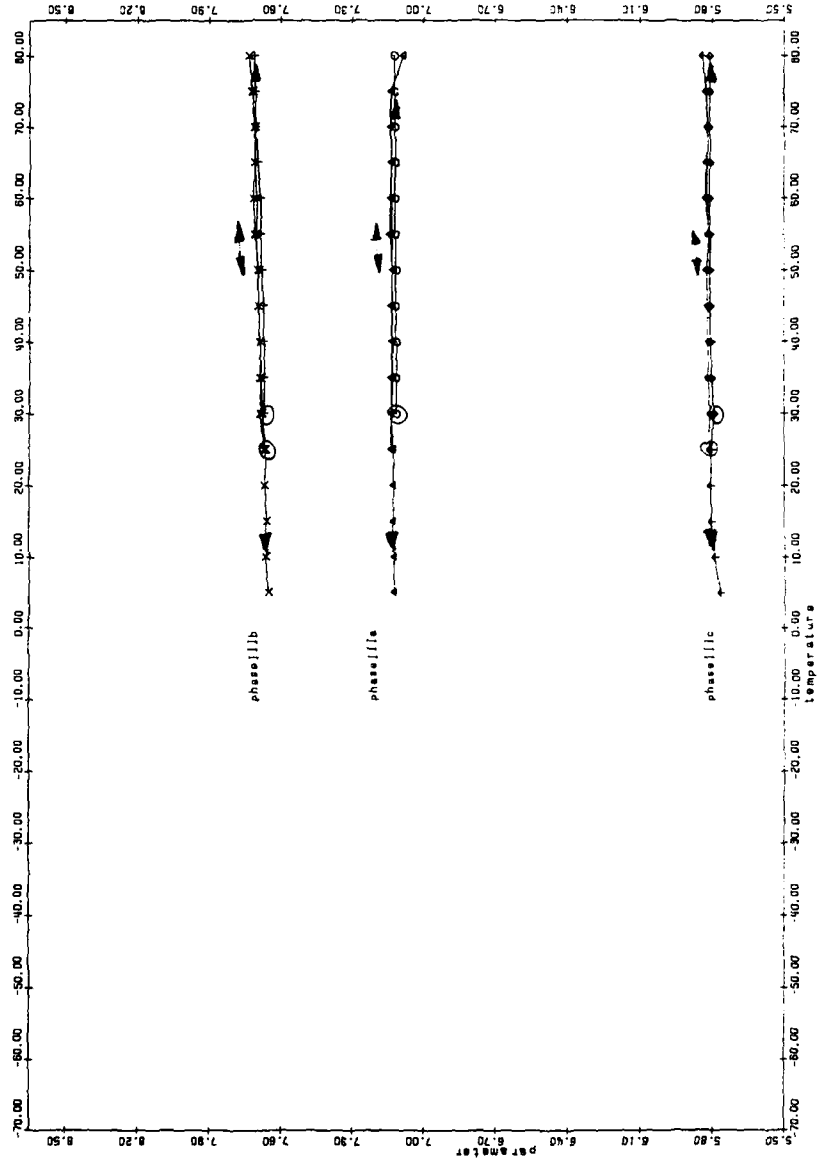
2% KF hum 20/80/-70/80



# Lattice Parameters

kf280988

2% KF hum 20/80/-70/80



# Lattice Parameters

kf280988

2% KF hum 20/80/-70/80

